

HEATING MONOCHLOROMETHANE IN AN AIRLESS PLACE AND AT A COLD TEMPERATURE PROCESS AND SELECTING A BINDING COMPONENT WITH A SELECTIVE CATALYTIC ACTIVITY AND A SELECTIVE CATALYST

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Annotatsiya: In the work, monochloromethane was selected for heating (at high temperature) in an air-absorbed environment. Methods of selecting a binding component for a catalyst that has a low catalytic activity and retains the characteristic of selective action are shown. Experiments were carried out in a catalyst bed reactor at temperatures of 400-500°C and a volumetric rate of monochloromethane of 450-2000 h⁻¹. In the process Mg-P-Zr-Fe/YukS-20; Mg-P-Zr-Fe/YukS-30 catalysts were used. In the Mg-P-Zr-Fe/YukS-20 sample, the conversion of monochloromethane was 66%, after 500 min, up to 42% of the monochloromethane conversion in Mg-P-Zr-Fe/YukS-20, Mg-P-Zr and in Fe/YukS-30 it decreased to 36%. Al₂O₃ was recommended to be used as a binder component, but the introduction of a binder led to a decrease in activity. The acidic properties of ammonia were studied using the method of thermoprogrammed desorption of ammonia. The regeneration process of the deactivated catalyst in the pyrolysis of monochloromethane was carried out using the method of combustion in air flow. Unreacted monochloromethane and reaction products were determined by the GSX method. In addition, the total selectivity of $\sum C_2-C_3$ in Mg-P-Zr-Fe/YukS-30 is 6.75 mol% higher than in Mg-P-Zr-Fe/YukS-20, and both Mg-P-Zr-Fe/ The activity of YukS-20 and Mg-P-Zr-Fe/YukS-30 catalysts decreases to 36.8% and 36.6% after 500 min of operation, which indicates the similarity in their deactivation. The purpose of the work is to create a bond for a slightly higher catalytically active and selective catalyst, which is separately isolated for the heating process of monochloromethane in an air-absorbed, i.e., vacuum environment, by slowly increasing the temperature. consists in choosing the constituent component.

Kalit so‘zlar: catalyst, temperature, pyrolysis, silicon aluminum magnesium phosphate, selective, ethylene, propylene, monochloromethane, reactor

INTRODUCTION

Experiments and experiments The amount of the catalytically active and selective catalyst and the binder in it has an effect on the process indicators (the chemical composition of the products and the conversion of monochloromethane) [1-3] .

The process of heating monochloromethane to ethylene and propylene in an air-absorbed environment, i.e. in a vacuum at a slightly higher temperature under the above conditions [4], allows obtaining industrially significant values of the main parameters of the process [5-8]. In order to increase the productivity of conversion of monochloromethane to ethylene and propylene as a result of pyrolysis, it is important to use a catalyst bed reactor that shows a slightly higher catalytic activity and has a selective effect [9-11].

During the testing of the YUKS-30 silicoaluminummagnesium phosphate fixed layer, it was found that with the increase in temperature, the catalyst selected for the process is sharply deactivated, which is accompanied by the large formation of methane [12-16].

Many studies have been carried out on the influence of the rate of feed volume on the parameters of the process of heating monochloromethane to low molecular weight unsaturated ethylene hydrocarbons in an airless space in pseudo-dilution mode[17-21]. During the process of heating monochloromethane, catalysts of the YKS type[22-26], as well as a catalyst with a slightly higher catalytic activity for heating monochloromethane in the absence of air and converting it to propylene and ethylene (pseudo-dilution mode) are deactivated due to the formation of carbon-retaining layers [27-31]. Catalytically active catalyst samples selected for the production of lower molecular weight ethylene series hydrocarbons by heating monochloromethane have been regenerated by oxidation in air flow to restore their initial activity [32-38].

EXPERIMENTAL PART

C₁-C₅ hydrocarbons in the gaseous reaction mixture was determined by the GSX method

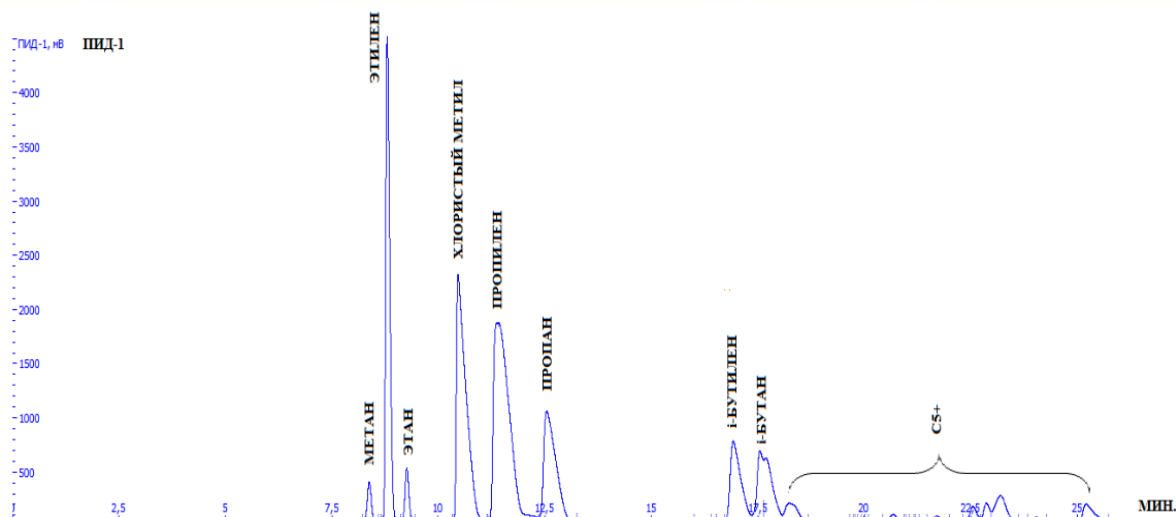


Figure 1. Chromatograms of pyrolysis products of methyl chloride Methodology for regeneration of deactivated catalysts based on high-silicon zeolite containing magnesium phosphate in silicomagnesium aluminum phosphate

Monochloromethane is heated at a slightly higher temperature and the catalyst air is sucked in for the synthesis of ethylene and propylene. Monochloromethane production is gradually stopped by injecting nitrogen into the reactor designed to heat monochloromethane and obtain propylene and ethylene. The temperature in the reactor designed to obtain

ethylene and propylene by heating monochloromethane in the absence of air rises to 550°C in a nitrogen flow.

At the end of the recovery process, an analysis of hydrogen chloride obtained by the alkalimetric method is carried out for the presence of absorbed hydrogen chloride.

Table 1

In silicoaluminummagnesium phosphates: Mg-P-Zr-Fe/YukS-20 and Mg-P-Zr-Fe/YukS-30 reaction product formation selectivity. Heat at 430°C, volume speed – 1500 h

EXPERIMENTAL RESULTS AND THEIR DISCUSSION

Catalyst samples	Tajriba time, min.	Hosil selectivity, mol%				
		CH ₄	C ₂ H ₄	C ₃ H ₆	$\sum C_2 - C_3$	C ₂₊
Mg-P-Zr-Fe/YukS-20	30	2.25	18.59	32,35	50.86	47.06
	60	2.26	27.78	42.99	70.79	27.13
	90	2.59	35,31	42.94	78.27	19,29
	500	4.11	40.01	40.88	81,87	14.11
Mg-P-Zr-Fe/YukS-30	30	2.18	17.06	31.07	48.08	49.94
	60	2.21	25.96	40.03	65,66	32,23
	90	2.46	30.98	40,44	71.35	26,29
	500	3.03	37.63	37,46	75.12	21.07

In the Mg-P-Zr-Fe/YukS-20 sample, the initial conversion of monochloromethane showed a high 66%. In both samples, it is observed that the activity of the catalyst selected for the process decreases gradually with the increase in the operating time of the catalyst

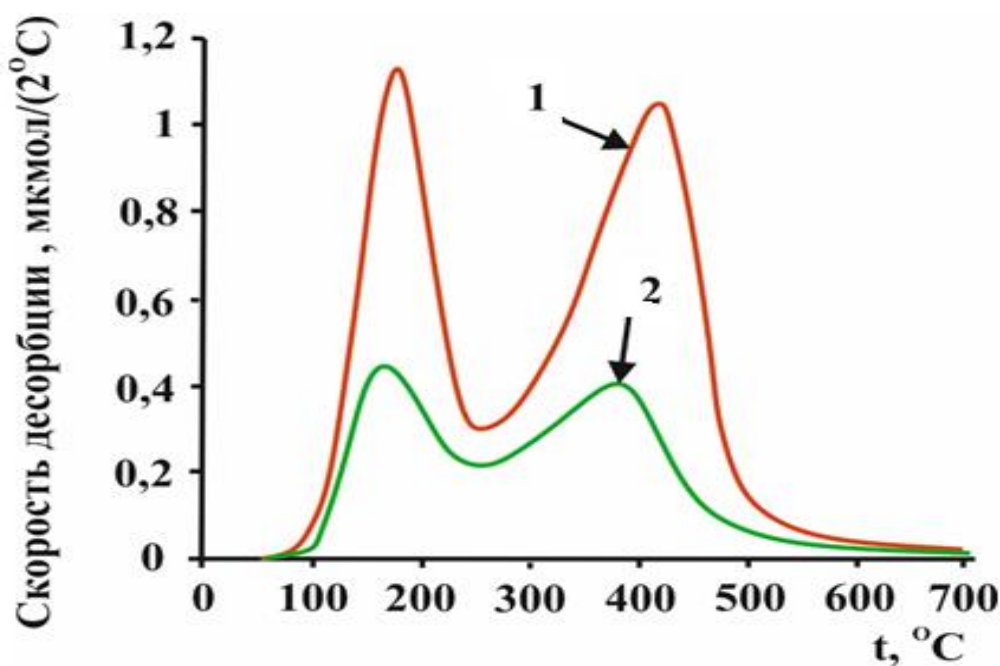
selected for the process. In Mg-P-Zr-Fe/YuKS-20, monochloromethane conversion decreases to 42% after 500 min from the start of the experiment, while in Mg-P-Zr-Fe/YuKS-30 it decreases to 36%.

In the Mg-P-Zr-Fe/YukS-20 and Mg-P-Zr-Fe/YukS-30 samples, the overall selectivity for low molecular unsaturated ethylene hydrocarbons, i.e. ethylene and propylene, is 500 minutes of selective catalyst operation. after that, it reaches 82 mol% and 75 mol% in the corresponding layer.

The selection of the ratio of the active component to the binding component is carried out by comparing the obtained data with the high results of monochloromethane in the absence of air in YuKS-30 - the pure active component. The amount of binding component was changed to 30-70 mass% in the selected catalyst exhibiting slightly higher catalytic activity and selective effect for the implementation of the process. The observed decrease in the conversion of monochloromethane in the catalysts selected for the progress of the process is related to the change in the physicochemical properties of YuKS-30 due to the composition of the binder and its granulation process. A comparison of the acidic properties of the pure active component - YKS and selective catalysts with catalytic activity allows to explain the observed decrease in their

activity. The study of acidic properties of the indicated samples was carried out using the method of thermoprogrammed desorption of ammonia (NH₃ TDD).

The granulation of YuKS-30 with a binding component does not cause a significant change in the quality composition of acidic centers, but it strongly affects their quantity per volume unit in comparison with YuKS-30-pure active ingredient. The catalytically active catalyst selected for the process is Mg-P-Zr-Fe/YukS-20 and pure active ingredient - YuKS-30, the TDD spectra presented in column 10 are characterized by the presence of two maxima. The two maxima present in the spectra correspond to two types of acid centers—weak centers with a maximum at 86°C and medium-strength centers with a maximum at 420°C. In both spectra, the positions of the maxima, which determine the strength of acidic centers, do not differ significantly. The quoted TDD spectra differ from each other in terms of the area under the TDD curve, which determines the amount of acidic centers per unit volume



1-clean YKS-30; 2-Mg-P-Zr-Fe/YukS-20

Figure 2. TDD spectra of silicon aluminum magnesium phosphates

According to the research results, the peaking process of YuKS-30 with binder does not cause changes in products and their quality composition. The main products are C₂-C₄ lower molecular unsaturated

ethylene and propylene, and the side products are C₁-C₅₊ paraffins. Mg-P-Zr-Fe/YukS-20, which exhibits slightly higher catalytic activity and selectivity, was chosen for the process. content on the catalyst, a

significant increase in the formation of ethylene is observed with a simultaneous decrease in the formation of propylene, but the increasing trend of their formation is maintained as the experiment continues. Binding component Al_2O_3 to 60 mass% in a selective catalyst with high catalytic activity leads to a maximum decrease of 5 mol% in ethylene formation. The selectivity of formation of lower molecular unsaturated ethylene hydrocarbons, i.e. ethylene and propylene (ethylene, propylene) in YuKS-30-pure active ingredient and YuKS-30 (40) is 82 mol% at 500 min of operation of the selected catalyst for the process taking place, the increase in the amount of binder leads to an insignificant decrease in the selectivity of lower

molecular unsaturated ethylene series hydrocarbons to 79 mol%.

Microspherical Mg-P-Zr-Fe/YukS-20 during high-temperature heating of monochloromethane in the absence of air testing a content-selective catalyst. Mg-P-Zr-Fe/YuKS-20 with a volume of 40 cm^3 for the heating process of monochloromethane in pseudo-dilution mode a batch of microsphere catalyst was prepared. Selective catalyst fraction with high catalytic activity is 0.06-0.12 mm. Mg-P-Zr-Fe/YukS-20 physicochemical properties of microsphere YuKS-30-pure active ingredient and Mg-P-Zr-Fe/YukS-20 are presented in comparison with the catalyst chosen to carry out the process (Table 2)

Table 2

Microspherical - Mg-P-Zr-Fe/YukS-20 and physico-chemical properties and compositions of the catalyst exhibiting somewhat higher catalytic activity.

No. t/p	Indicator	Unit of measure	YUKS-30 is a pure active ingredient	Mg-P-Zr-Fe/YukS-20	Microspherical Mg-P-Zr-Fe/YukS-20
1	Amount of shabasite phase	%	100	60	60
2	The amount of binder	% mass	-	40	40
3	Surface surface	m^2/g	606	521	531
4	Pore size	sm^3/g	0.31	0.33	0.33
5	The amount of acidic centers per unit volume	$\mu\text{mol/g}$	1850	1500	1115

The information given in table 2 is from the article apparently It turns out that the adsorption surface of YuKS -30 catalysts with Al_2O_3 is much smaller than that observed in the pure active component of YuKS-30 . The binding component partially blocks the micropores of YuKS-30, which means that the specific surface area of the selectively acting catalysts bound with Al_2O_3 remains lower than that of the active component. A decrease in the amount of centers with an acidic property is also associated. Mg-P-Zr-Fe/YukS-20 Mg-P-Zr-Fe/YukS-20 with a fixed layer in the microsphere in the pseudo-liquefaction mode allows the conversion of monochloromethane to increase compared to the conversion that occurs at. From the data given in Table 2, it can be seen that the tested samples of catalysts with slightly higher catalytic activity selected for carrying out the process with Al_2O_3 , having exactly the same composition, show almost the same physico-chemical

characteristics. It is Mg-P-Zr-Fe/YukS-20 It is a reason to assume that the catalytic properties of the microsphere and the Mg-P-Zr-Fe/YuKS catalyst are compatible. Since both test samples have the same composition and almost the same physico-chemical properties, this method of carrying out the process of monochloromethane at a slightly higher temperature and in a vacuum, i.e. in a vacuum: a fixed catalyst layer or requires similarity in the catalytic ability of the transfer regardless of the mode of forgery. Mg-P-Zr-Fe/YukS-20 Mg-P-Zr-Fe/YukS-20 in the fixed layer of the process in the false dilution mode in the microsphere allows an increase in monochloromethane conversion compared to the observed conversion.

Investigation of the influence of this temperature on the process of heating monochloromethane in the pseudo-dilution mode at a slightly higher temperature in which air is sucked in, that is, in a vacuum. A series of

experiments were carried out at a temperature in the range of 400-500°C and a volume injection rate of monochloromethane of 1000 h⁻¹ for the parameters of the process of heating monochloromethane at a slightly balanced temperature in pseudo-dilution mode (Fig. 3). Selective volumetric injection of monochloromethane is immobile, has high catalytic activity, and in the

selective catalyst bed, in the range of 1000-1500 h⁻¹ volumetric injection, monochloromethane conversion and lower molecular unsaturated ethylene series hydrocarbons, i.e. ethylene and propylene, are with selectivity of 60-80 mol% and 70-81 mol%, respectively.

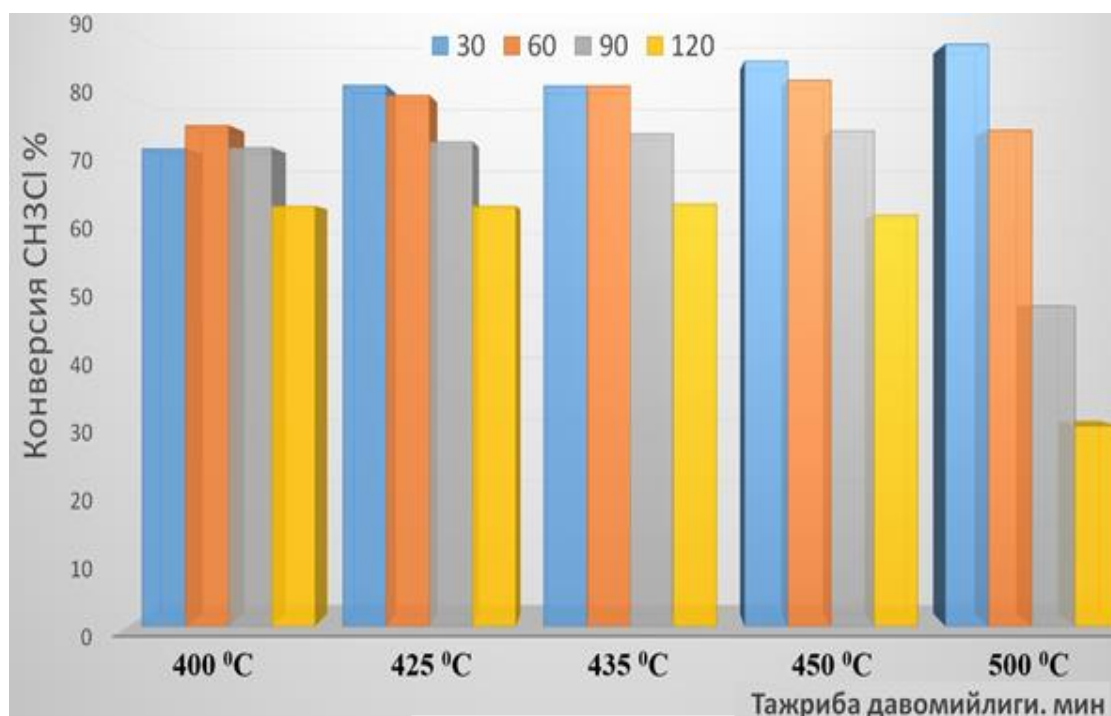


Figure 3. Dynamics of monochloromethane conversion in the temperature range of 400-500°C

Increasing the temperature from 400°C to 500°C allows the initial conversion of monochloromethane to increase from 73% to 87%. It was shown that increasing the temperature during the experimental period caused a sharp deactivation of the catalyst selected for the process. Therefore, a sharp decrease in monochloromethane conversion is observed during 500 minutes of operation of the catalyst selected for the process at 475°C, which exhibits a slightly higher catalytic activity.

The analysis of how much the selectivity of ethylene and propylene production changes after 500 minutes of operation of the active catalyst, which exhibits slightly higher catalytic activity and has a specific selective effect, at different temperatures, allows to determine certain laws. will give. With an increase in temperature in the range of 400-500°C, the formation of ethylene increases by 15-20 mol% on average during the experimental period. As the temperature increases, the selectivity of propylene formation also increases during the experimental period,

but the lower the temperature, the more significant this increase. Thus, the selectivity for propylene with high catalytic activity is selected to carry out the process at 400°C, and during the operation of the selective catalyst for 500 min, the selectivity on propylene increases from 23 mol% to 47 mol%, to carry out the process (at 450°C) during 90 min of operation of the selected catalyst, a small increase from 27 mol% to 38 mol% is observed, and during the next 30 min, the selectivity for propylene decreases to 36 mol%. Increasing the temperature to 475 °C caused the selectivity of ethylene and propylene formation to drop from 28 mol% to 22 mol% and from 27 mol% to 2.5 mol% respectively during 500 min of operation of the catalytically active and selective catalyst. cause.

For the implementation of the process, the selective catalyst exhibiting slightly higher catalytic activity and increasing the temperature from 400°C to 500°C during the first 30 minutes of the operation of the selective catalyst is 33 mol% of the total selectivity of the formation of C₂-C₃, respectively. leads to a

gradual increase from to 55 mol%. After 500 minutes of operation of the catalyst selected for the process, if its activity decreases, the total selectivity of the formation of C₂-C₃ unsaturated ethylene series hydrocarbons passes through the maximum and is 77 mol% and corresponds to the temperature range of 430-450°C. Mg-P-Zr-Fe/YuKS-20 is also suitable for heating monochloromethane in a microsphere at high temperature without air. The selectivity of methane formation increases from 11 mol% to 46 mol% as the time of the experiment at 475°C temperature increases, and it changes within 2-3 mol% limits at 425°C temperature.

Investigation of the influence of the feed rate of feedstock on the process of heating monochloromethane at high temperature and in the absence of air in pseudodilution mode. Reducing the monochloromethane volume rate from 2000 to 450 h⁻¹ at peak temperature of 450°C resulted in the expected significant increase in monochloromethane conversion. At the indicated contact time, monochloromethane conversion increased from 15.0% to 77.0% after 500 min of operating the slightly higher catalytically active catalyst selected for the process. An increase in temperature resulted in a sharper decrease in monochloromethane conversion over the studied range of monochloromethane volumetric feed rates. Thus, high-temperature decomposition of monochloromethane in the absence of air at high temperatures is less

efficient, due to the excessive consumption of the initial raw material. At a higher temperature of 450°C, the selectivity to ethylene is slightly higher than that at 430°C by 6-9 mol% on average. By increasing the volume of monochloromethane from 450 to 2000 h⁻¹, the selectivity for ethylene at 430 °C increases from 27.0 mol% to 43.0 mol%, and at 450 °C it increases from 33.0 mol% to 45. increases to 0 mol%. It was found that the change in the selectivity of ethylene formation in the range of 450-2000 h⁻¹ of feed rates per volume unit at both temperatures is similar. The volume rate of monochloromethane from 450 to 2000 h⁻¹ in the temperature range of 430-450°C is selected for the process. with leads to an overall increase in propylene production from 68 mol% to 85 mol%.

CONCLUSION

Thus, summarizing the experimental results shows that the conversion of monochloromethane in the range of 1000 to 1500 h⁻¹ of monochloromethane volumetric rate at a temperature of 430°C is maintained in the range of 52.0-71.0%, the total selectivity of propylene formation with ethylene is 77- It is stable at 79 mol%. The activity of selective catalysts with slightly higher catalytic activity, which were selected for the progress of the process, reached almost the same value during 500 min of operation: Mg-P-Zr-Fe/YuKS-20 in the microsphere it decreases to 36.8% and in Mg-P-Zr-Fe/YuKS-20 to 36.6%, which indicates the similarity of their deactivation laws

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