

# Study of Some Kinetic Laws of Methyl Monochloride Pyrolysis Reaction

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**Abstract.** Purpose of work-is to study some kinetic laws of the heating decomposition reaction of monochloromethane in an airless environment at a slightly higher temperature. The growing demand for unsaturated ethylene series hydrocarbons in recent years shows how important the work in this field is. Pyrolysis of methyl chloride was carried out in a special flow and differential reactor. YuKS-20/30, YuKS-20, YuKS-30 and silicoaluminum-magnesium phosphates: YuKS-30 Si, YuKS-30 Fe, YuKS-30, and YuKS-30 modified with Mg were used as catalysts. The selectivity and acidic properties of modified YuKS-30, YuKS-20/YuKS-30, YuKS-20 catalysts were studied using the thermoprogrammed desorption method of ammonia. Changes in CH<sub>3</sub>Cl conversion dynamics were observed at different temperatures and catalysts. From the obtained results, the selectivity for propylene in YuKS-20/YuKS-30 and YuKS-20 samples was 32mol% and 35mol%, and in YuKS-30 it was 44mol%. In YuKS-20 and YuKS-20/YuKS-30 samples, the total selectivity of C<sub>2</sub>-C<sub>3</sub> formation at 500 min is 61 mol%, and in YuKS-30 it is equal to 71 mol%. Monochloromethane conversion was 80.5%. In the temperature range of 400-500°C, the activity of silicoaluminummagnesium phosphate was 77-84%, the active period of the catalyst increased at the volume rate of monochloromethane of 1000 h<sup>-1</sup>. Ethylene extraction selectivity increases from 26 mol% to 41 mol% after 500 min at a volume rate of 1000-1500 h<sup>-1</sup>, and propylene selectivity increases to 45-47 mol%.

## 1 Introduction

At least a dozen large petrochemical products are produced on the basis of ethylene and propylene. In addition, we must admit that methods and equipment have been developed for converting methane to methyl chloride in various ways and obtaining various other products from methane and its products [1-6]. According to the amount of production of low molecular weight ethylene hydrocarbons, it is possible to know the level of development of the petrochemical industry in the country[7-13]. In turn, the production of lower molecular ethylene series hydrocarbons is one of the fastest growing branches of the petrochemical industry[14-19]. One of the main considered methods for the production of low molecular weight ethylene series hydrocarbons is steam and catalytic cracking processes used in industry[20-23]. The reaction of formation of lower molecular ethylene hydrocarbons of monochloromethane is based on the method of their production by chemical processing of natural gas [24-27]. The proposed method of processing natural gas into ethylene and

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propylene through the intermediate formation of monochloromethane makes it possible to create a more economically and technologically useful process[28].

It is known from the conducted studies that the selective catalyst selected for the decomposition of monochloromethane at high temperature in silicon aluminum phosphate YuKS-30 is observed to decrease its activity as a result of the formation of carbon-retaining layers [18-21]. The process of decomposition of monochloromethane in silicoaluminophosphate YuKS-30 is followed by rapid carbonization of the catalyst selected to carry out the process leading to its passivation. Recovery of the initial activity of silicoaluminophosphate is carried out by incineration in air flow at a temperature of 550°C[29-32].

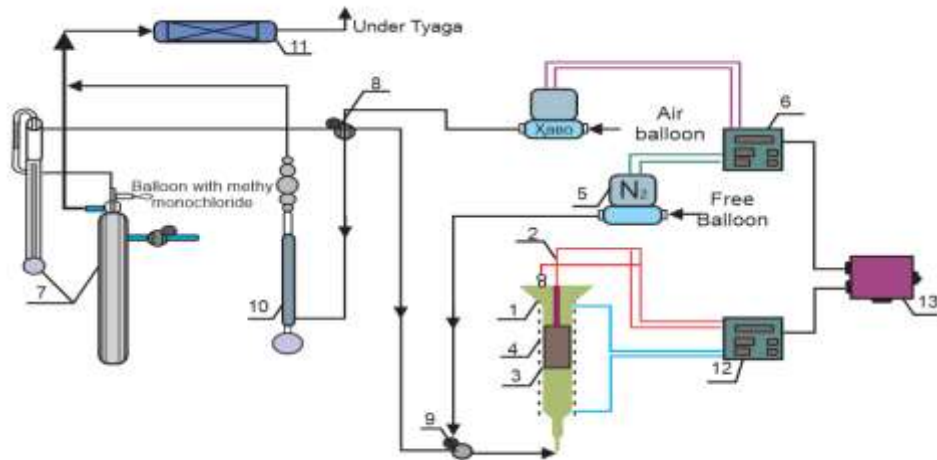
## 2 Experimental Part

During the start and stop of the process, the laboratory was flushed with nitrogen. Reactor (1) prepared for obtaining lower molecular ethylene hydrocarbons as a result of decomposition of monochloromethane at slightly higher temperatures is heated to a certain level and its cooling is carried out by supplying gaseous nitrogen. Nitrogen is sent from a cylinder to the reactor designed to obtain ethylene and propylene by heating monochloromethane at a high temperature. Nitrogen consumption is provided by the RRG-10 regulator (5), its control is carried out by the control unit (6). Monochloromethane is sent from the cylinder (8) and through the three-way valve (9) to the reactor prepared for obtaining propylene and ethylene, heating monochloromethane to a certain degree. The specified volume of monochloromethane is set using the "manostat-rheometer" (7) dosing system. The reactor designed to heat monochloromethane and obtain ethylene and propylene consists of 1 tube made of simax glass. Monochloromethane enters from the bottom of reactor 1 through Schott filter 3. The reactor is equipped with 3 thermowells for obtaining low molecular weight ethylene series hydrocarbons by decomposing monochloromethane at a slightly higher temperature in a special environment with air intake. An electrical coil (obmotka) 4 made of steel wire was used to heat the reactor designed to heat monochloromethane and obtain ethylene and propylene. The temperature regime in the reactor designed for decomposition of monochloromethane is carried out with the help of 12 integral-differential thermoregulators, 2 controlled thermocouples installed in the sleeve of the reactor designed for extraction. Temperature measurement accuracy is  $\pm 1^\circ\text{C}$ . When the temperature in the reaction medium rises to 500°C, the TRM-148 device has a light and sound alarm, as well as a block that ensures the shutdown of 4 electrical circuits from the network (220 V voltage).

A reactor with a selected catalyst layer is used to heat monochloromethane and obtain ethylene and propylene in conducting tests on the selection of an active component based on YKS. The cross-sectional surface of the reactor designed to obtain ethylene and propylene by heating monochloromethane at high temperature is 3.3 cm<sup>2</sup>. The volume of the selected catalyst for heating monochloromethane and obtaining propylene and ethylene is 3-5 cm<sup>3</sup>. The remaining volume is filled with a porcelain nozzle. The location of the selected catalyst for the reactor designed to produce propylene and ethylene by heating monochloromethane was determined by the previously imaged temperature section of the reactor designed to decompose 1 monochloromethane.

Experimental studies of the activity of the selected catalyst for the decomposition of microspherical monochloromethane are carried out in pseudodilution mode, which is created by passing the initial monochloromethane through a 3 Schott filter. The cross-sectional surface of the reactor designed for airless decomposition of monochloromethane was 3.3 cm<sup>2</sup>. The volume of the loaded catalyst with high catalytic activity is 10.5-50 cm<sup>3</sup>. Reaction gases 1 from the reactor enters the Drexel beaker. Hydrogen chloride is absorbed from the gaseous

mixture of reaction products in the Drexel glass. The volume of the Drexel glass is 500 ml. The hydrochloric acid solution is neutralized with alkali and disposed of.



**Fig. 1.** A laboratory device designed to produce propylene and ethylene by decomposing methyl chloride in an air-entrained atmosphere at a slightly higher temperature.

After the start of the experiment, it is first waited for 30 minutes and analyzed, then the analysis is carried out after another 30 minutes, this process is repeated, that is, it is analyzed every 30 minutes. Technological conditions (temperature, linear velocity, volumetric saturation velocity, recovery mode) for decomposition of monochloromethane at a slightly higher temperature by choice recommendations are based on the results of several test experiments. The selected catalyst recovery rate for monochloromethane decomposition varied from 100 to 1000 cm<sup>3</sup>. The pressure in the reactor designed to decompose monochloromethane is 1-3 atm.

### 3 Experimental Results and Discussion

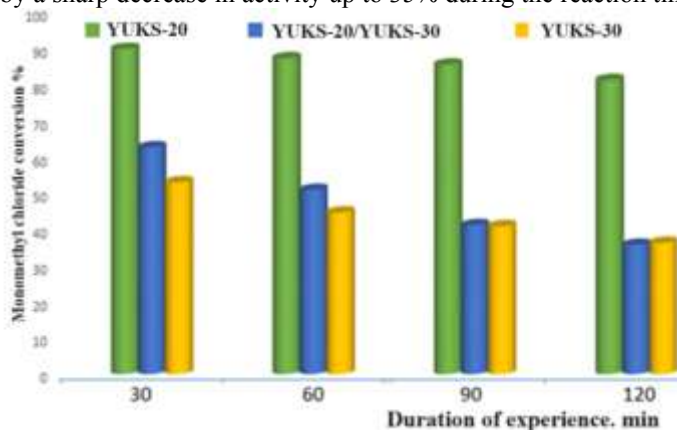
The total selectivity of formation of lower molecular unsaturated ethylene hydrocarbons reaches 81-85 mol.% after 500 minutes of operation of silicoaluminummagnesium phosphates: YuKS-30<sup>Si</sup>, YuKS-30<sup>Fe</sup>, YuKS-30. In the YuKS-30 sample modified with Mg, the value of this multi-layer is 70 mol.% (Table 1).

**Table 1.** Selectivity of formation of methyl chloride pyrolysis products in modified silicoaluminummagnesium phosphates. Temperature 425°C. Volume speed 1200 h<sup>-1</sup>.

Substance	Experiment time, min.	YUKS-20	Modified catalysts based on YuKS-20		
			YUKS-20 <sup>Si</sup>	Yuks-20 <sup>Fe</sup>	YUKS-20 <sup>Mg</sup>
Formation selectivity mol%					
CH <sub>4</sub>	30	1.75	1.42	1.46	1.57
	60	1.03	1.15	1.15	1.86
	90	1.07	1,20	1,31	1,40
	120	1.13	1.57	1.79	3.39
C <sub>2</sub> H <sub>4</sub>	30	19.57	19.52	18.77	24.25
	60	23.33	31,91	31,11	29,30
	90	30,30	38,71	37,94	29,10
	120	34.72	44.18	42.07	27.79

$C_3H_6$	30	27.55	39.62	38.29	37.05
	60	38.17	50,10	48.38	40.69
	90	46.01	45.15	44.34	40.32
	120	46.58	41.45	39.94	41.66
$\Sigma C_{2=C_3=}$	30	47,10	59,11	57.04	61.27
	60	61,50	81.99	79.46	69.97
	90	76.29	83.83	82.27	69.39
	120	81.27	85.62	81.99	69.44
$\Sigma C_{2+}$	30	51,21	39.52	41.54	37.22
	60	37.49	16.93	19.44	28,21
	90	22.68	15.03	16.47	28.26
	120	17.65	12.84	16.26	27.24

As can be seen from the obtained data, the modification of silicoaluminummagnesium phosphates based on YuKS-30 leads to a decrease in monochloromethane conversion, which affects the overall selectivity of the formation of  $C_2-C_3$  unsaturated ethylene series hydrocarbons, that is, propylene with ethylene. does not show. The silicoaluminummagnesium phosphates used in these experiments differ greatly in the type of crystal lattice structure, as well as in the size of the crystals. The crystal lattice structure of YuKS-20 with large-sized crystals creates a 52% lower value of the initial conversion of monochloromethane. A cocrystallized with a crystal size comparable to that of YuKS-30 provides an intermediate value for initial conversion of monochloromethane YuKS-20/YuKS-30. The studied samples of silicoaluminummagnesium phosphates: YuKS-20 and YuKS-20/YuKS-30 are characterized by a sharp decrease in activity up to 35% during the reaction time (Fig. 2).



**Fig. 2.** In silicoaluminummagnesium phosphates: YuKS-20, YuKS-20/ YuKS-30, YuKS-30 information on the conversion of monochloromethane. Temperature 430°C, volume speed 500 hours l

The selectivity of the formation of ethylene, as well as  $C_{2+}$ , almost does not depend on the type of crystal lattice structure. The values for all three samples are close and vary between 25-31 mol.% for ethylene and 27-35 mol.% for  $C_{2+}$  after 500 min of experiment. The selectivity for propylene obtained in YuKS-20/YuKS-30 and YuKS-20 samples is significantly lower than the value of this indicator obtained in YuKS-30. So, after 500 minutes of exploitation of silicoaluminummagnesium phosphates, the selectivity of formation of propylene in YuKS-20/YuKS-30 and YuKS-20 was 32mol% and 35mol%, respectively, and in YuKS-30 it was 44mol%. It was observed that the selectivity of the total formation of  $C_2-C_3$  in 500 minutes of the experiment in YuKS-20 and YuKS-20/YuKS-30 samples did not exceed 61 mol%, in YuKS-30 this indicator was equal to 71 mol%. The

formation of methane in YuKS-20/Yuks-30 and YuKS-20 silicoaluminummagnesium phosphates was two times higher than the data obtained for YuKS-30 (Table 2).

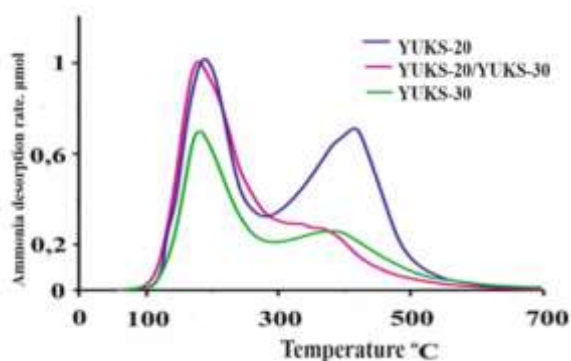
**Table 2.** Selectivity of formation of products of high temperature heating of monochloromethane based on silico- aluminomagnesium phosphates. Temperature 430°C, Volume speed = 500 h<sup>-1</sup>.

Substance	Test time, min.	YUKS-30	YUKS-20	YUKS-20/30
		Formation selectivity, mol.%		
CH <sub>4</sub>	30	2.68	2.79	2,81
	60	2.01	3.42	3.11
	90	1.60	3.86	3.33
	500	1.44	3.96	4.02
C <sub>2</sub> H <sub>4</sub>	30	10.58	18.24	20.69
	60	16.66	22.79	24.56
	90	22.04	25.66	26.69
	500	26.18	25.64	31.22
C <sub>3</sub> H <sub>6</sub>	30	22.29	32.47	34.38
	60	32.84	36.29	36.89
	90	39.45	37.53	38.23
	500	44.88	35.82	32.17
∑ C <sub>2=</sub> – C <sub>3=</sub>	30	32.86	50.68	55.07
	60	49.49	59.09	61.44
	90	61.48	63.19	64,92
	500	70.99	61.45	63.37
∑ C <sub>2+</sub>	30	64,51	46.56	42.18
	60	48.53	37.55	35.49
	90	36.96	32.99	31.83
	500	27.67	34.65	32.66

Samples per volume unit and the strength of the center of mass differ from each other sufficiently. YuKS-30 shows the power of maximum concentration and acidity centers. It is known that acidic centers enable the formation of propylene with unsaturated ethylene series hydrocarbons.

**Table 3.** Silicoaluminummagnesium phosphates: YuKS-30, YuKS-20/Yuks-30, YuKS-20 acid properties research data using the thermoprogrammed desorption method of ammonia.

Silicoaluminum magnesium phosphate	Amount of acidic centers per unit volume, mmol/g
YUKS-20	830
YUKS-20/ YUKS-30	1080
YUKS-30	1640



**Fig 3.** Silicoaluminummagnesium phosphates: YuKS-20, YuKS-20/YuKS-30, YuKS-30 spectra of ammonia obtained using thermoprogrammed desorption method

Evaluation of catalytic properties of YuKS-20, YuKS-20/ YuKS-30, YuKS-30 showed that sample YuKS-30 is considered the most promising.

According to the results of the research work, it was found that the selective catalyst selected for the decomposition of monochloromethane in silicoaluminummagnesium phosphate YuKS-30 has a decrease in its activity as a result of the formation of carbon-retaining layers.

According to the results of the research, the decomposition process of monochloromethane in silicoaluminummagnesium phosphate YuKS-30 is observed with rapid carbonization of the catalyst, which leads to its passivation. Recovery of the initial activity of silicoaluminummagnesium phosphate is carried out by burning in air at a temperature of 550°C.

A number of experiments on the stability of YuKS-30 silicoaluminummagnesium phosphate under the conditions of continuous switching of "reaction-regeneration" cycles were carried out, and the regeneration process was carried out by the method of combustion in air flow.

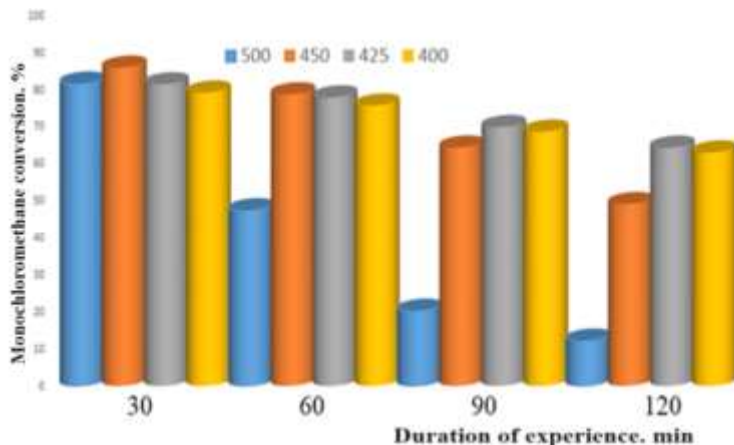
The selected catalyst was initially carbonized by heating monochloromethane at a temperature of 430°C and a volume rate of 1500 h<sup>-1</sup> for 500 min. The regeneration was fed at a temperature of 550°C and a volumetric rate of 350 h<sup>-1</sup> for 500 min .

Reaction: Temperature 430°C, volume speed-1500 h<sup>-1</sup> , experiment duration 500 min.

Regeneration : Temperature 550°C, volume speed-350 hours<sup>-1</sup> · duration of regeneration 135 minutes.

In the new sample, the initial conversion of monochloromethane was 80.5%, after 15 cycles - 79.9%. The obtained data, the selected methodology and conditions of the regeneration process of the catalyst selected for the implementation of the deactivated process allow to completely restore its catalytic properties. Repeated regeneration process does not reduce its catalytic activity, which speaks of its stability both during the process of heating monochloromethane and under the selected conditions of the regeneration process.

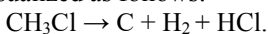
Study of the influence of feed volume rate and temperature on the catalytic decomposition of monochloromethane in the presence of silicoaluminummagnesium phosphate and its parameters. Figure 4 shows the results obtained from determining the temperature dependence of the conversion of monochloromethane and the selectivity of the formation of lower molecular ethylene series hydrocarbons. Initially, silicoaluminummagnesium phosphate was tested using a fixed bed reactor with a selective catalyst at a flow rate of 1500 h<sup>-1</sup> of monochloromethane at a temperature range of 400-500°C. The results are presented in Figure 4.



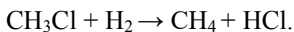
**Fig. 4.** Change dynamics of CH<sub>3</sub>Cl conversion at different experimental times in YuKS-30 silicoaluminum magnesium phosphate at different temperatures . Volume speed - 1500 hours<sup>-1</sup>.

Of 400-500°C is 77-84% (Fig.4). In the temperature range of 400-450°C, the conversion of monochloromethane slows down as the duration of the experiment increases. At 500°C, monochloromethane conversion decreases from 84% to 10%. It is possible that the increase in temperature allows secondary conversion reactions of intermediates to condensation products. This helps to increase the formation of carbon storage layers, their accumulation in silicoaluminummagnesium phosphate leads to the blocking of active centers, thereby deactivating YuKS-30.

The subsequent transformation of intermediate compounds into condensation products and their carbon storage layers leads to the formation of hydrogen in the reaction products of decomposition of monochloromethane at a slightly higher temperature in the air-entrained environment. The brittle-reaction of hydrogen formation during the catalytic decomposition of monochloromethane can be visualized as follows:



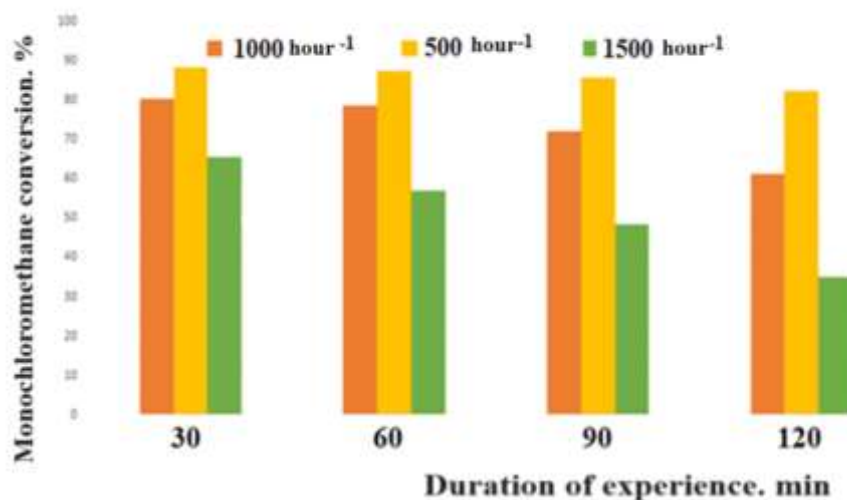
the p- arching reaction interacts with monochloromethane and methane is obtained:



Thus, the appearance of methane in the reaction products is associated with the formation of carbon storage layers, which cause the deactivation of the catalyst selected for the process. It follows that with an increase in the temperature of the process, rapid deactivation of the catalyst selected for the process is observed through the formation of a large amount of methane.

The general selectivity for hydrocarbons of the lower molecular ethylene series (ethylene, prorylene) is maintained at 81 mol.% with increasing temperature in the range of 400-450 °C , increasing the reaction temperature to 500 °C causes a sharp decrease in the indicator value to 28 mol.% is observed with 500°C are approximately due to a sharp increase in the formation of carbon-retaining compounds, which block the active sites and thereby lead to the formation of unsaturated ethylene series hydrocarbons, i.e., propylene with ethylene. prevents.

Determination of the influence of feed rate on the conversion of monochloromethane and the selectivity of the formation of low molecular weight unsaturated hydrocarbons. Investigating the influence of the volume rate of monochloromethane supply on the process parameters. The immobilized and selective catalyst has a very high catalytic activity and selectivity in YKS-30 silicoaluminummagnesium phosphate using a reactor. was carried out. The results are presented in Figure 5 and Table 4.



1-volume speed-1500 hours<sup>-1</sup> ; 2-volume speed 1000 h<sup>-1</sup> ; 3-volume speed 500 h<sup>-1</sup> ;  
 Temperature-430°C

**Fig. 5.** The dynamics of monochloromethane conversion at different volume rates of reagents in YuKS-30 with the duration of the experiment

From Figure 5, it can be seen that reducing the volume rate of monochloromethane to 1000 h<sup>-1</sup> allows to increase the period of active operation of the selected selective catalyst. Thus, at a volumetric rate of injection of monochloromethane of 1000 h<sup>-1</sup> the activity of YuKS-30 decreases imperceptibly from 89% to 80% during the 500 h<sup>-1</sup> experiment. The increase in the volumetric rate allows to accelerate the overall rate of the monochloromethane transformation reaction, at the same time, to accelerate the process of formation of carbon storage layers and their accumulation in silicoaluminummagnesium phosphate, which in turn causes the deactivation of YuKS-30.

**Table 4.** The influence of the volumetric rate of monochloromethane on the selectivity of the formation of reaction products in silicoaluminummagnesium phosphate YuKS-30 the experience is 500 minutes. The temperature is 430°C.

	volume velocity, hour <sup>-1</sup>		
	500	1000	1500
Formation selectivity, mol.%			
CH <sub>4</sub>	1.49	1.12	1.66
C <sub>2</sub> H <sub>4</sub>	26.26	34.76	41.08
C <sub>3</sub> H <sub>6</sub>	44.84	46.58	45.77
∑ C <sub>2</sub> – C <sub>3</sub>	71.07	81.28	86.77
C <sub>2+</sub>	28.03	17.67	11.69

The data on the selectivity of ethylene indicate that increasing the load on silicoaluminummagnesium phosphate due to the increase in the volumetric rate of monochloromethane in the interval of 1000-1500 hours<sup>-1</sup> increases the selectivity of silicoaluminummagnesium phosphate to 26 mol of ethylene after 500 minutes of operation. % to 41 mol.%. In this case, the selectivity for propylene remains stable at the level of 45-47 mol.% in all studied ranges of monochloromethane volumetric velocities. The overall selectivity for lower molecular weight ethylene series hydrocarbons increases the volumetric



rate of monochloromethane from 1000 h<sup>-1</sup> to 1500 h<sup>-1</sup> from 71 mol.% to 87 mol.% after 500 min.

## 4 Conclusion

Thus, the selectivity of C<sub>2+</sub> hydrocarbons formation decreases by a factor of two with the increase in volumetric rate under the conditions of increase in selectivity and decrease in monochloromethane conversion. According to the data of previous physical and chemical studies of carbonized silicoaluminummagnesium phosphate YuKS-30, its deactivation and reduction of micropores per volume unit are observed during the decomposition of monochloromethane. YuKS-30 and YuKS-30 silicoaluminummagnesium phosphates were found to be analogues. In this case, increasing the volumetric rate of monochloromethane activity of YuKS-30 in the range of 1000-1500 h<sup>-1</sup> leads to the narrowing of silicoaluminummagnesium phosphate cellular channels, and small molecules such as ethylene and propylene are formed in them. From the results of the research, it is possible that the increase in the temperature of decomposition of monochloromethane, as well as the volume load on the catalyst selected for the process, leads to a sharp deactivation of silicoaluminummagnesium phosphate YuKS-30, but an increase in selectivity to ethylene is observed.

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