

Laws of Catalytic Aromatization Reaction of C₁-C₄-Carbohydrates and Texture Characteristics of Catalysts

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Abstract--- For the catalytic aromatization reaction of lower alkanes (MoO₃)_x · (ZnO) in the catalytic system based on the study of the promoter properties of Zr and B (MoO₃)_x · (ZnO)_y · (ZrO₂)_z · (B₂O₃)_k catalyst was selected and this catalyst studied some kinetic laws of the catalytic aromatization reaction of C₁-C₄-hydrocarbons. As a result of research, the content of propane is 98.2%; iso-butane-0.18%; butane-1.62% proved that the reaction yield and selectivity relative to aromatic hydrocarbons had the highest value when the mixture was catalytic aromatized, and at this time the conversion of the mixture was 95.4%, the yield of aromatic hydrocarbons was 66.5% and the relative selectivity of aromatic hydrocarbons was 69.7%. The texture characteristics of the catalysts obtained in the study are also given.

Keywords--- Petroleum Gases, Propane, Butane, Conversion, Selectivity, Yield, Catalyst.

I. INTRODUCTION

The aromatization of lower alkanes is one of the topics that attract the greatest attention of both industry and specialists studying the fundamental problems of catalysis [1-5]. The greatest successes in this direction are associated with the use of zeolites [6-9]. They are characterized by the presence of an ordered channel structure and increased thermal stability. The hydrogen form of these zeolites contains Brønsted and Lewis acid centers, which allows them to be used as catalysts, alternative to mineral acids.

This approach is promising as a way of qualified processing of gas hydrocarbon raw materials (including associated petroleum gas) into valuable petrochemical products. A detailed study of the mechanism of aromatization of associated petroleum gas led to the conclusion that it is necessary to operate acid and base centers in the catalyst as vicinal groups [10-15]. In this context, it is of interest to introduce additives into the composition of the catalyst that can not only change the activity of the catalyst, but also affect its structure.

However, the practical implementation of the conversion of associated petroleum gas to aromatic hydrocarbons on zeolite catalysts is constrained by a number of factors. Significant disadvantages of zeolite aromatization catalysts include the blocking of zeolite micropores by carbon deposits and the occurrence of side cracking reactions with the formation of methane [16-24], leading to inappropriate use of raw materials. The deactivated zeolite catalyst is freed from coke deposits using an oxidative regeneration (annealing) procedure, which, in turn, can lead to the destruction of active sites and catalyst structure. To overcome the aforementioned disadvantages, a number of approaches are used, including the introduction of promoters and the creation of an additional mesoporous structure

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in the zeolite matrix. The introduction of promoters allows one to control the acid properties of zeolite and significantly affects the behavior of zeolites in catalysis [25-29].

Based on the above, the aim of the work is to create stable catalysts with high catalytic activity, productivity and selectivity, characterized by a decrease in the degree of coking to obtain aromatic hydrocarbons by catalytic aromatization of petroleum gases.

II. EXPERIMENTAL PART

High silica zeolite (VCC) synthesized by the hydrothermal method was used as the initial catalyst. The starting components in the synthesis were kaolin of the composition: 73% SiO₂, 7.2% Al₂O₃, 6% Na₂O, 13.8% H₂O. Hexamethylenediamine was used as a structure-forming component. To transfer the zeolite to the active H form, it was treated with a 25% aqueous solution of NH₄Cl at 90 ° C for 2 h.

After removal of sodium cations, the zeolite in the NH₄ form was dried at 110 ° C and calcined in air at 550 ° C for 6 h. Zinc was introduced by impregnation of the zeolite with an aqueous solution of zinc nitrate (Zn (NO₃)₂ · 6H₂O). The concentration of zinc (in terms of metal) in the catalyst was 2.0% of the mass.

The acidic properties of the Zn-containing zeolite were studied by temperature-programmed desorption of ammonia. The strength of the acid sites of the catalyst was estimated from the temperature maxima on the thermal desorption curve, and their concentration was determined by the amount of ammonia desorbed at the moment of fixing the desorption peaks, and was expressed in μm per 1 g of catalyst.

The specific surface of the Zn-VCC and its porous structure parameters were determined by low-temperature nitrogen adsorption using the Sorbtometer M automatic gas adsorption analyzer. The specific surface area of the test sample was calculated using the multipoint method of Brunauer, Emmett, and Teller (BET). The volume and pore size of the catalyst were determined using the BJH model (Barett – Joyner – Halenda) from isothermal adsorption and desorption data at a relative pressure of P / Po = 0.99.

High resolution transmission electron microscopy (HRTEM) images were obtained with JEM 2010 and JEM-2200FS electron microscopes (JEOL Ltd.) with a lattice resolution of 0.14 and 0.1 nm, respectively.

Energy dispersive X-ray (EDX) spectra were obtained in scanning mode with the JEM-2200FS in dark field mode (HAADF-STEM) using the built-in JED-2300T spectrometer. Quantitative element analysis was done using EDX spectrometer software. Samples were prepared as suspensions in ethanol on Cu-support grids, followed by air drying. The conversion of propane (degree of purity 99.95% vol.), Butane (degree of purity 99.2% vol.) Of the propane-butane fraction into aromatic hydrocarbons (ArU) was studied on a flow-through bench apparatus with a stationary catalyst bed at atmospheric pressure, reaction temperature 450 –600 ° C and the volumetric feed rate of the feedstock 1000 h – 1.

The analysis of gaseous and liquid reaction products was carried out by GLC using a Chromatek-Crystal 5000.2 chromatograph.

To determine the composition of the liquid phase, we used a BP-1 PONA capillary column (100 mx 0.25 mm x 0.5 μm), and to determine the composition of the gas phase, we used a capillary column GS-Gas-Pro (60x0.32 mm) and a packed Carbosieve S-II (4 mx 2 mm, 60/80 mesh) columns. During the experiments, the degree of conversion

of the starting lower alkanes, the yield of gaseous and liquid products were determined, and the selectivity of the formation of reaction products was calculated. The conversion of the feedstock and the selectivity of the formation of reaction products were calculated using data from a chromatographic analysis of the composition of the feedstock, gaseous and liquid reaction products, as well as data on their quantitative yield.

III. EXPERIMENTAL RESULTS AND THEIR DISCUSSION

The thermodynamic probability of aromatization of lower alkanes is high, with ethane being converted to aromatic hydrocarbons at temperatures above 600°C, propane above 500°C, and butanes above 400°C. The aromatization selectivity and high yield of lower alkanes in modified zeolites depends on the nature and concentration of the modifier, the catalyst preparation method, the location of the metals, and the catalyst processing conditions.

The effect of temperature in the presence of various catalysts on the yield of target products of aromatization reaction of petroleum gases was studied. The results obtained are presented in Table 1 below.

Table 1: Influence of Various Promoters Added to the Reaction Product $(\text{MoO}_3)_x \cdot (\text{ZnO})_y$ on the Aromatization of Petroleum Gases

Catalyst	Temperature of the experiment, °C				
	490	500	510	520	530
C ₄ H ₁₀ in gas phase, % mass					
$(\text{MoO}_3)_x \cdot (\text{ZnO})_y$	9.56	7.81	4.81	6.85	2.98
$(\text{MoO}_3)_x \cdot (\text{ZnO})_y \cdot (\text{ZrO}_2)_z$	9.44	9.41	6.88	4.73	4.62
$(\text{MoO}_3)_x \cdot (\text{ZnO})_y \cdot (\text{ZrO}_2)_z \cdot (\text{B}_2\text{O}_3)_k$	8.88	7.63	5.82	4.73	3.42
Content of aromatic hydrocarbons, % mass					
Content of toluol, % mass					
$(\text{MoO}_3)_x \cdot (\text{ZnO})_y$	26.38	27.50	28.42	31.00	32.87
$(\text{MoO}_3)_x \cdot (\text{ZnO})_y \cdot (\text{ZrO}_2)_z$	29.20	30.50	33.10	34.50	36.50
$(\text{MoO}_3)_x \cdot (\text{ZnO})_y \cdot (\text{ZrO}_2)_z \cdot (\text{B}_2\text{O}_3)_k$	27.34	28.48	30.31	30.05	34.26
Content of xylol and ethyl benzol, % macc					
$(\text{MoO}_3)_x \cdot (\text{ZnO})_y$	33.24	33.17	30.07	28.61	26.27
$(\text{MoO}_3)_x \cdot (\text{ZnO})_y \cdot (\text{ZrO}_2)_z$	35.20	33.30	30.60	28.20	28.70
$(\text{MoO}_3)_x \cdot (\text{ZnO})_y \cdot (\text{ZrO}_2)_z \cdot (\text{B}_2\text{O}_3)_k$	36.51	32.48	30.81	28.53	28.57
Content of benzol, % mass					
$(\text{MoO}_3)_x \cdot (\text{ZnO})_y$	6.23	6.88	8.12	10.49	12.66
$(\text{MoO}_3)_x \cdot (\text{ZnO})_y \cdot (\text{ZrO}_2)_z$	6.99	7.86	8.96	10.86	13.78
$(\text{MoO}_3)_x \cdot (\text{ZnO})_y \cdot (\text{ZrO}_2)_z \cdot (\text{B}_2\text{O}_3)_k$	6.88	7.78	9.28	10.76	10.89

As can be seen from the table, the optimum temperature for the conversion of petroleum gases is 520-530°C. We then continued the experiments in the presence of a catalyst containing $(\text{MoO}_3)_x \cdot (\text{ZnO})_y \cdot (\text{ZrO}_2)_z \cdot (\text{B}_2\text{O}_3)_k$. The results obtained are presented in Table 2.

Table 2: Catalytic Transformation of Petroleum Gases in a Catalyst Containing $(\text{MoO}_3)_x \cdot (\text{ZnO})_y \cdot (\text{ZrO}_2)_z \cdot (\text{B}_2\text{O}_3)_k$

Composition of raw material, mass	Conversion and productivity of products, mass %	Reaction temperature, °C		
		500	520	530
Ethane-3.0 Propane-40.6 I-buthane-18,9 Buthane-37,5	Conversion	78.6	90.7	90.7
	Gases	90.4	77.4	72.9
	Liquid	33.8	42.5	59.8
	Selectivity on AU yield,%	42.5	47.8	65.8
Propane-7.4 I-buthane-20.1 Buthane-72.5	Conversion	80.8	87.2	93.6
	Gases	89.2	72.7	64.8
	Liquid	44.8	51.8	62.7
	Selectivity on AU yield,%	56.3	67.2	67.0
Propane-98,2 I-buthane-0,18 Buthane-1,62	Conversion	63.2	67.8	95.4
	Gases	89.6	78.2	72.8
	Liquid	50.4	55.8	66.5
	Selectivity on AU yield,%	81.6	83.6	69.7

As can be seen from Table 2, the content of propane-98.2; iso-butane-0.18; when a mixture containing butane-1.62 is catalytic aromatized, the reaction yield and selectivity relative to aromatic hydrocarbons are at their highest. At this time, the conversion of the mixture is 95.4%, the yield of aromatic hydrocarbons is 66.5% and the relative selectivity of aromatic hydrocarbons is 69.7%.

Table 3: Catalytic Transformation of Petroleum Gases in a Catalyst Containing $(\text{MoO}_3)_x \cdot (\text{ZnO})_y \cdot (\text{ZrO}_2)_z \cdot (\text{B}_2\text{O}_3)_k$

$T_{op}, ^\circ\text{C}$	520	530	540	560	580	600
W, c^{-1}	1000	800	1000	1000	800	1000
Conversion,%	82.7	100	94.6	89.8	100	98.8
$\sum prod. AU, \%$	57.8	69.8	62.4	53.4	54.8	58.6
AV Selectivity,%	70.1	69.8	66.0	59.5	54.8	59.3
Liquid catalyst composition, % mass						
Benzol	22.9	28.5	23.8	23.8	31.0	28.9
Toluol	34.6	41.5	35.4	39.2	43.5	33.1
Ethyl benzol	21.8	12.8	16.2	16.8	7.8	9.5
Xylol	8.5	6.5	8.5	8.59	5.9	7.8
C_{8+}	6.9	4.2	6.4	3.9	1.9	3.2
Naphtalines	9.6	7.5	12.6	11.6	8.8	18.5
Gas catalyst composition, %						
Carbon	2.1	2.0	2.0	1.67	2.1	1.6
Ethane	17.4	48.0	38.2	14.2	49.8	44.4
Ethylene	13.5	17.8	18.7	22.2	16.3	20.6
Propane	17.5	Sl	12.4	18.7	Sl	1.8
Propylene	15.5	Sl	10.8	21.2	Sl	5.6
Buthanes	7.8	-	-	1.1	-	-
C_4 -olephines	3.7	-	-	3.1	-	-

Figure 1 shows the dependence of the selectivity of the formation of alkanes and aromatic hydrocarbons on the conversion of propane to $(\text{MoO}_3)_x \cdot (\text{ZnO})_y \cdot (\text{ZrO}_2)_z \cdot (\text{B}_2\text{O}_3)_k$ -WCC at various process temperatures.

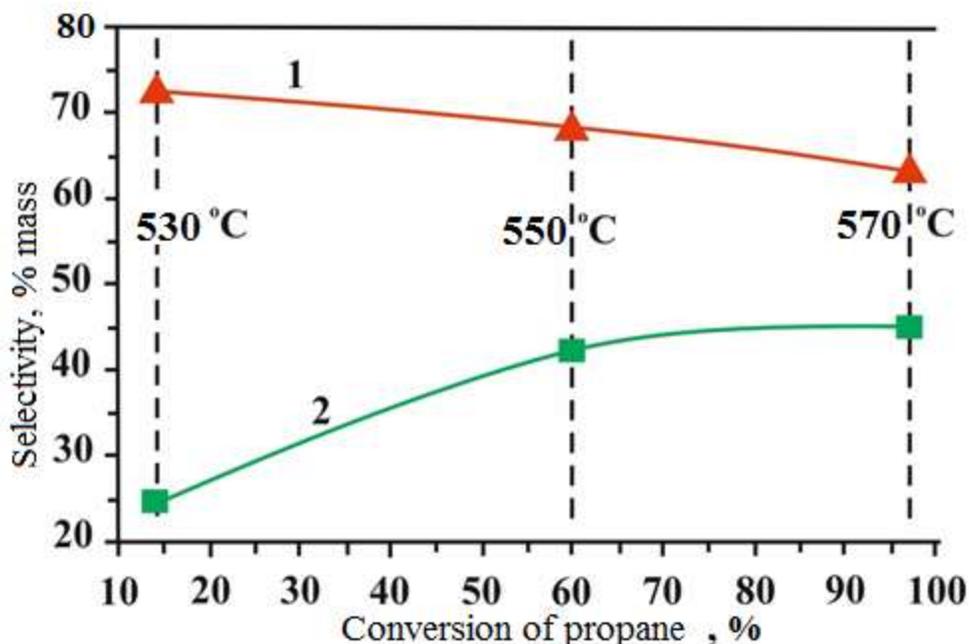


Figure 1: The Dependence of the Selectivity of the Formation of Alkanes and Aromatic Hydrocarbons on the Conversion of Propane to $(\text{MoO}_3)_x (\text{ZnO})_y (\text{ZrO}_2)_z (\text{B}_2\text{O}_3)_k$ -WCC at Various Process Temperatures

With an increase in the reaction temperature, there is an increase in the degree of conversion of propane and the selectivity of the formation of aromatic hydrocarbons, which at 930°C reach 96% and 45.1% respectively. In gaseous reaction products, with increasing reaction temperature, the selectivity of alkane formation decreases. The results of studies on the conversion of propane and butane to $(\text{MoO}_3)_x (\text{ZnO})_y (\text{ZrO}_2)_z (\text{B}_2\text{O}_3)_k$ -VCC are shown in Table. 4.

Table 4: The Indicators of the Process of Conversion of C_3, C_4 Alkanes to $(\text{MoO}_3)_x (\text{ZnO})_y (\text{ZrO}_2)_z (\text{B}_2\text{O}_3)_k$ -VCC

Feedstock	T, °C	X%	A%	Product selectivity, % mass				
				H ₂	CH ₄	Alkanes C ₂ -C ₅	Alkenes C ₂ -C ₅	Arenas
Propane	400	4	0.7	3.8	12.8	50.1	14.6	18.9
	450	14	2.1	8.2	17.1	45.5	14.4	14.8
	500	60	19.3	4.0	19.7	38.7	5.4	32.2
	530	96	44.0	3.0	23.3	36.8	1.9	45.8
	550	100	41.0	3.6	28.8	26.2	2.1	41.1
Butane	450	13	0.9	2.3	4.8	76.9	11.5	4.7
	500	40	17.6	2.8	7.6	37.8	8.1	43.7
	520	79	30.8	2.9	13.9	37.7	6.7	38.7
	530	100	49.5	2.9	18.6	36.0	2.8	49.5
PBF	400	6	0.8	5.3	10.3	54.9	17.4	12.1
	450	17	2.9	7.7	18.6	42.1	14.0	17.6
	500	59	26.7	3.9	15.4	30.5	5.2	45.1
	530	95	42.4	3.2	20.8	35.4	2.3	44.6
	550	100	42.9	3.5	26.0	26.0	2.1	42.3

Note. T-process temperature, X-conversion, A-yield of aromatic hydrocarbons

As seen from the above data, a significant conversion of propane starts at a reaction temperature 4 to 50 ° C. With increasing temperature, the conversion of propane increases and reaches 100% at 55 ° C. At the same time, the selectivity of methane formation increases (from 12, 8 to 2 8, 8 %). The formation of aromatic hydrocarbons begins at 500 ° C, and their maximum yield (4 4, 0%) is achieved at 530 ° C. It should be noted that the composition of aromatic hydrocarbons is strongly influenced by the process temperature. With its increase increases the formation of benzene, methylene n- and dimethylnaphthalenes, reduced concentration of xylenes and alkylbenzenes C₈₊ and toluene content passes through a maximum at a temperature of 550 ° C. Butane undergoes aromatization much easier than propane.

The conversion of butane to $(\text{MoO}_3)_x(\text{ZnO})_y(\text{ZrO}_2)_z(\text{B}_2\text{O}_3)_k$ -VCC begins already at 400 ° C, and a noticeable formation of aromatic hydrocarbons starts at 450 ° C. With an increase in the process temperature, the conversion of butane increases, and at 530 ° C it reaches 100%. The yield of aromatic hydrocarbons under these process conditions is 4 9.5%, a close amount of aromatic hydrocarbons is formed during the conversion of propane, but only at a temperature of 55 ° C. A noticeable conversion of PBP, as in the case of propane, begins at a reaction temperature of 450°C. At the same time, it is seen that $(\text{MoO}_3)_x(\text{ZnO})_y(\text{ZrO}_2)_z(\text{B}_2\text{O}_3)_k$ -WCC during aromatization of PBP exhibits a higher overall and aromatizing activity at the initial temperatures of the process, that is, at 450– 500 ° C. With an increase in temperature to 550 ° C and higher, the yield of aromatic hydrocarbons in the case of the conversion of PBP slightly differs from the yield of aromatic hydrocarbons observed in the aromatization of propane. Thus, we can conclude that under comparable conditions the degree of conversion of the initial hydrocarbon feed and the yield of the target product formed on $(\text{MoO}_3)_x(\text{ZnO})_y(\text{ZrO}_2)_z(\text{B}_2\text{O}_3)_k$ -VCC, the higher the pain - Chez length of the hydrocarbon chain alkane. Consequently, to achieve a certain level of conversion and yield of aromatic hydrocarbons using butane as feedstock will require a low order - perature than in the case when subjected to propane conversion. In fig. Figure 2 shows the data on the conversion of C₃, C₄ alkanes to $(\text{MoO}_3)_x(\text{ZnO})_y(\text{ZrO}_2)_z(\text{B}_2\text{O}_3)_k$ -WCC at different process temperatures. It can be seen that 100% butane conversion is achieved at a temperature of 550 ° C, while propane and PBP are achieved at 600 ° C.

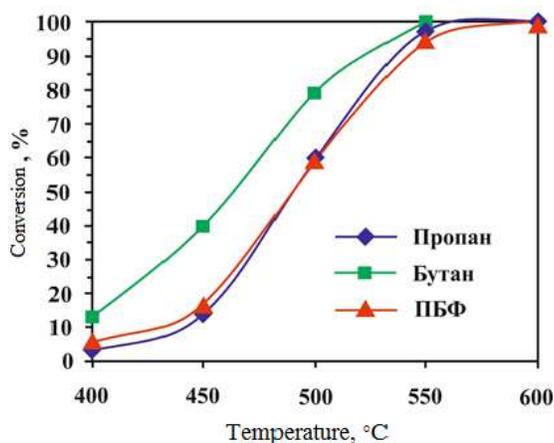


Figure 2: A TED for the Conversion of C₃, C₄ Alkanes to $(\text{MoO}_3)_x(\text{ZnO})_y(\text{ZrO}_2)_z(\text{B}_2\text{O}_3)_k$ -VKTS Process at different Temperatures

The results of determining the phase composition of the studied catalysts by x - ray diffractometry before and after participation in the catalysis are shown in Fig. 3 - 6.

The position of characteristic peaks in all diffractograms was typical of zeolites. The desilicate zeolite (see Fig. 3) also retains its structure, but the intensity of characteristic peaks decreased, which is associated with a partial destruction of the zeolite structure.

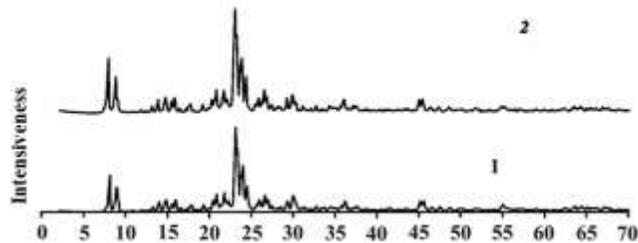


Figure 3: X-ray diffraction Patterns of $(\text{MoO}_3)_x(\text{ZnO})_y(\text{ZrO}_2)_z(\text{B}_2\text{O}_3)_k$ -VCC before (1) and after (2)

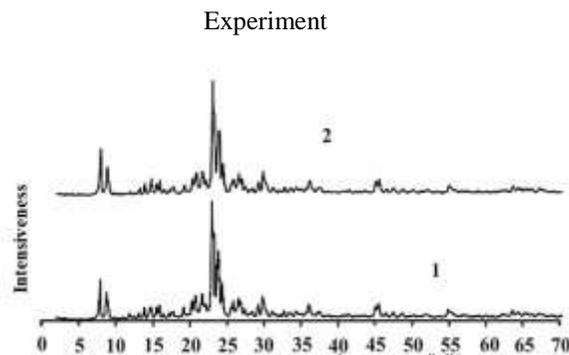


Figure 4: Diffractogram $(\text{MoO}_3)_x(\text{ZnO})_y(\text{ZrO}_2)_z(\text{B}_2\text{O}_3)_k$ -VKTS : before (1) and after (2) Experiment

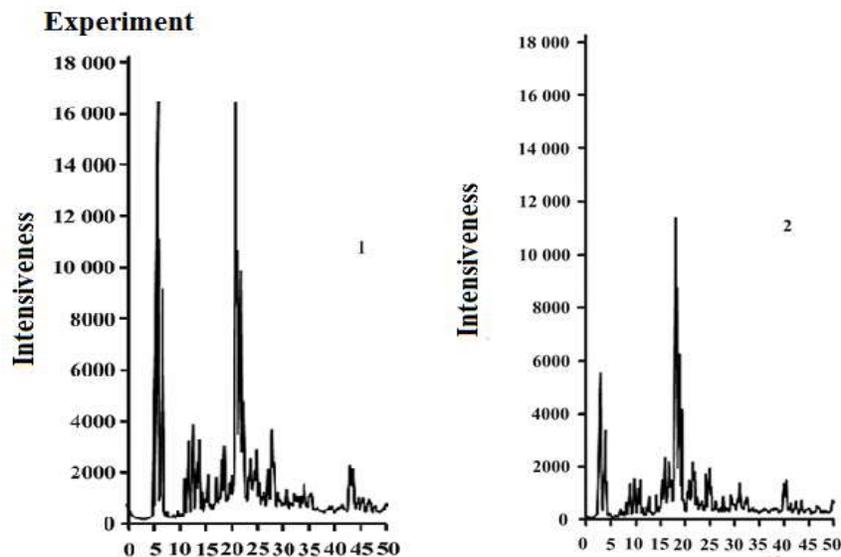


Figure 5: X-ray diffraction Patterns of $(\text{MoO}_3)_x(\text{ZnO})_y(\text{ZrO}_2)_z(\text{B}_2\text{O}_3)_k$ -VCC before (1) and after (2) Desilicate

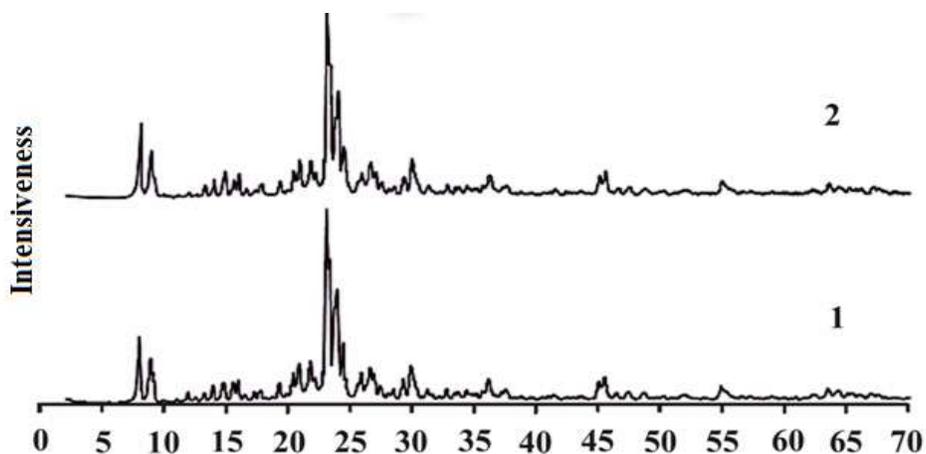


Figure 6: X-ray Diffraction Patterns of $(\text{MoO}_3)_x (\text{ZnO})_y (\text{ZrO}_2)_z (\text{B}_2\text{O}_3)_k$ -VCC Desilicatisir before (1) and after (2)

Experiment

Table 5 below shows the texture characteristics of the synthesized catalysts.

Table 5: Specific Surface Area and Porosity of the Catalyst

№	Catalyst	$S_{\text{sol.}} \text{ m}^2 / \text{g}$	$V_{\text{pore.}} \text{ cm}^3 / \text{g}$
1	$(\text{MoO}_3)_x (\text{ZnO})_y (\text{ZrO}_2)_z$	178.6	0.456
2	$(\text{MoO}_3)_x (\text{ZnO})_y (\text{B}_2\text{O}_3)_z$	191.5	0.476
3	$(\text{MoO}_3)_x (\text{B}_2\text{O}_3)_y (\text{ZrO}_2)_z$	194.3	0.498
4	$(\text{MoO}_3)_x (\text{ZnO})_y (\text{ZrO}_2)_z (\text{B}_2\text{O}_3)_k$	214.7	0.518

IV. CONCLUSION

Thus, a catalyst containing $(\text{MoO}_3)_x \cdot (\text{ZnO})_y \cdot (\text{ZrO}_2)_z \cdot (\text{B}_2\text{O}_3)_k$ was selected for the catalytic aromatization reaction of lower alkanes. In the presence of this catalyst, some kinetic laws of the catalytic aromatization reaction of C_1 - C_4 -hydrocarbons were studied. As a result of research, the content of propane is 98.2%; iso-butane-0.18%; butane-1.62% proved that the reaction yield and selectivity relative to aromatic hydrocarbons had the highest value when the mixture was catalytic aromatized, and at this time the conversion of the mixture was 95.4%, the yield of aromatic hydrocarbons was 66.5% and the relative selectivity of aromatic hydrocarbons was 69.7%.

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