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INFLUENCE OF TECHNOLOGICAL PARAMETERS ON CATALYTIC AND REGENERATION PROPERTIES OF CoHZSM-SO_4^{2-}/ZrO_2 COMPOSITE SYSTEM OF LOW-TEMPERATURE ISOMERIZATION OF STRAIGHT-RUN GASOLINES

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The composite catalyst of 10% ZrO_2, 0.4% Co, 2% SO_4^{2-}/HZSM-5 designed for low-temperature conversion of straight-run gasoline (SRG) was synthesized. The influence of technological parameters (temperature, volumetric rate and ratio of hydrogen to initial raw material - H_2/SRG) on catalytic and regeneration properties of the catalyst was investigated and optimal process conditions under which the conversion of C_{8+} is 76.8%, and the sum of hydrocarbons C_5-C_6, consisting of 78.8% of high-octane isomers, rises from 11.0% to 61.1.2%, were established. Regenerative characteristics of the catalyst were studied. The stability of the work of the samples and the amount of carbon compounds were determined depending on the duration of the reaction, the temperature of preliminary reduction of the samples by hydrogen, and the introduced hydrogenolysis metals (Co, Ni). It found that rise in the regeneration temperature of a 10% ZrO_2,0.4% Co, 2% SO_4^{2-}/HZSM-5 sample from 380 to 500^0C leads to the stability of the catalyst. Replacing of Co with Ni enhances the stability of the catalyst due to the decrease in the rate of accumulation of carbon deposits; however, the selectivity of the catalyst is reduced.

Keywords: *straight-run gasoline, composite catalyst, technological parameters, regeneration, isomers, hydrocarbon*

INTRODUCTION

Analysis of the literature shows that the interest in the process of isomerization, including straight-run gasoline, due to the particular topicality of raising their octane characteristics, has not fallen in recent decades [1-6-5]. However, despite the sufficient amount of work to convert straight-run gasoline, a question of their direct involvement in isomerization processing remains practically not illuminated and is limited to studies into C5-C6 n-paraffins isolated from their composition with subsequent involvement into isomerization processing [7,8-6,7]. We have shown in [9,10- 8,9] the results of the direct involvement of straight-run gasoline into the low-temperature isomerization-disproportionation process on composite Me, HZSM /SO_4^{2-}/ZrO_2 systems. Also, the possibility of replacing high-temperature dehydrocyclization treatment (reforming) by isomerization (isoriforming) and changes in the hydrocarbon composition of the feedstock were established depending on the nature of zeolite and the amount of SO_4^{2}-.

It is topicald to study the effect of process parameters on activity changes and regeneration characteristics of 10% ZrO_2, 0.4% Co, 2% SO_4^{2-}/HZSM-5 catalyst in the course of conversion of straight run gasoline. Owing to the fact that the process under study proceeds at low temperatures, it is not thermodynamically complicated by the dehydrogenation process. The stability of the catalyst in the process of work is identified

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through the accumulation of carbon deposits while the earlier studies showed that their accumulation occurs as a result of bimolecular interaction of the components of straight run gasoline with products of their hydrocracking. In this case, the formation of C_{11} and C_{11} intermediate paraffin is realized at temperatures lower than their boiling points, and as a result they are accumulated on the catalyst blocking the access of the reactant.

Consequently, in this case, the success in performing the task of stabilizing the functioning of catalysts lies in the decomposition of such an intermediate compounds. As is known, coke is a carbonaceous compound with varied content of carbon and hydrogen. In terms of the process, their accumulation is limited by two factors - hydrocracking or hydrogenolysis. The problem of cleavage of the formed high molecular paraffin whose accumulation leads to deactivation of the catalyst, was resolved by the introduction of hydrogenolysing components. In this case the elements of group VIIIB are most active.

Thus, the aim of this work was to examine the influence of technological parameters on the activity of isomerization-disproportionation catalysts of straight-run gasoline; on the dependency of stability of samples and the amount of the formed carbon compounds; on the duration of the reaction; on the temperature of preliminary reduction of the samples by hydrogen, as well as the introduction of hydrogenolytic metals (Co, Ni).

EXPERIMENTAL PART

The object of the study was composite catalysts prepared by modifying HZSM-5 zeolite with cobalt, nickel and zirconium. In order to add hydrogenolysing properties to the catalyst and stabilize the process, nickel was introduced into the zeolite component. Modification of the initial zeolites was carried out by ion-impregnating introduction of cobalt or nickel ions, as well as impregnation with a solution of sulfating agent $(NH_4)_2SO_4$ counting on the quantity of ions.

The salts of $ZrO\ (N0_3)_2 \cdot 2H_20$, $NiN0_3$, $CoSO_4{\cdot}7H_20$ were used as a source of active components for the preparation of catalysts. The zeolites modified by the methods described above were molded using a binder component - an alumina hydrogel based on 25 g per 100 g of dry catalyst weight.

After carefully mixing the modified zeolites with hydrogel, the resulting mass was molded by pressing through a spinneret with a cross section of 1.2-1.5 mm, air dried up for 24 hours under an electric lamp, and then granulated into cylinders 4.5 mm high. The molded samples were dried up for 4 hours at 353 K and 383 K and calcined at 623 K (2 h) and 823 K (4 h).

Thus, the composite systems of M-20 - 0.4% Ni/HZSM-5/SO_4^{2-} (2%) -ZrO_2 (10%) /Al_2O_3 (25%) and M-11 0.4% Co/HZSM-5/SO_4^{2-}(2%)-ZrO_2 (10%) / Al_2O_3 (25%) were synthesized.

Following the use of raw material, a gasoline fraction of the composition%: C_4 (1.0)) iso-C_5 (1.0), n-C_5 (1.2), iso-C_6 (5.0), n-C_6 (3.8), iso-C_7 (22.0), and C_7 (6 , 5) and the remainder is a mixture of higher-molecular alkanes C_{8+} (59.5).

The conversion of this mixture was studied through the use of a flow-type laboratory catalytic installation equipped with a quartz reactor. The volume of catalyst loaded into the reactor varied within 1-5 sm^3. The reaction products for the analysis were taken straight after the reaction and placed into receivers cooled by dry ice.

The products of the reaction were analyzed on an Auto System XL gas chromatograph (PerkinElmer) and a LXM-80MD chromatograph with a flame-ionization detector.

To study the effect of hydrogen, all other conditions being equal, the hydrogen supply changed from 10 to 60 ml/min, which made it possible to change the ratio H_2/CH from 1 to 6, where CH is an average molecular weight of straight-run gasoline (SRG) taken after calculation of the sum of hydrogen atoms to carbon atoms.

The determination of sums of carbonaceous deposits accumulating on the catalysts was carried out by oxidative treatment. Before the experiments the unit was purged with an inert gas (He) (1 hour). The catalyst was then treated with air at the rate of 4 l/hr at 500°C. The amounts of CO_2 and H_2O released were determined by their absorption by anhydrone and ascaride placed in appropriate capsules, followed by gravimetric analysis of these ampoules, and directly by analysis of the carbon deposits which formed as result of reactions, using the element analyzer of the firm "TruSpec Micro". It should be noted that, in order to avoid errors, the air entering the reactor was also passed through tubes filled with anhydrone and ascaride.

RESULTS AND DISCUSSIONS

As is known, the main technological parameter of the reaction is the temperature of its realization. Fig.1 says that the process of SRG conversion is satisfactorily realized in the temperature range 140-200^{0}C.

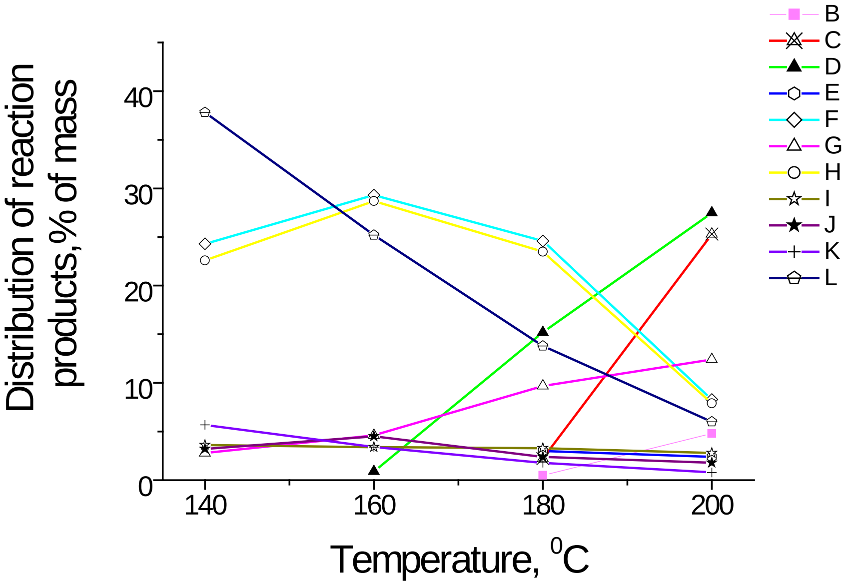

Fig. 1. Influence of temperature on the distribution of products of the conversion of straight-run gasoline (SRG) on catalyst M-11.
$H_2/CH = 3$, volumetric rate = 2.5 h^{-1}, composition of initial raw materials: C_1-C_2 = 0; C_3 = 0; i-C_4 =0; C_4 = 1; i-C_5 =1; C_5 = 1.2; i-C_6 =5 ; C_6=3.8; i-C_7= 22 ; C_7 = 6.5; C_{8+}= 59.5
B - C_1-C_2; C - C_3; D- i-C_4; E - C_4; F- i-C_5; G - C_5 ; H - i-C_6; I- i-C_7; J- C_6; K - C_7; L - C_{8+}

The process course at a temperature of 200°C, as can be seen from Fig.1, results in the consumption of hydrocarbon raw materials to form undesirable gaseous alkanes. A drop in the temperature up to 140^{0}C results in the loss of catalyst activity. Hence, isomerization-disproportionation processing of SRG is most suitable to take place within the temperature range of 160- 180°C.

Fig.2 summarizes the catalytic data for catalyst M-11. As can be seen from these data, when the straight-run gasoline contacted with the M-11 composite catalyst within the temperature range of 140-180^{0}C high-molecular-weight C_{8+} components are converted with high catalyst activity, a fairly high selectivity was observed in the accumulation of C_5-C_6 alkanes where the content of isomeric constituents lies within the limits 88.0-78.8% and in this time the conversion of C_{8+} paraffin was 36.4 -76.8%.

The distribution of the catalytic products is dependent on the volumetric feed rates of the reactant. Table 1 shows the dependence of the conversion of C_{8+} paraffin and the yield of C_4 alkanes on the volumetric rate of SRG feed into the reaction.

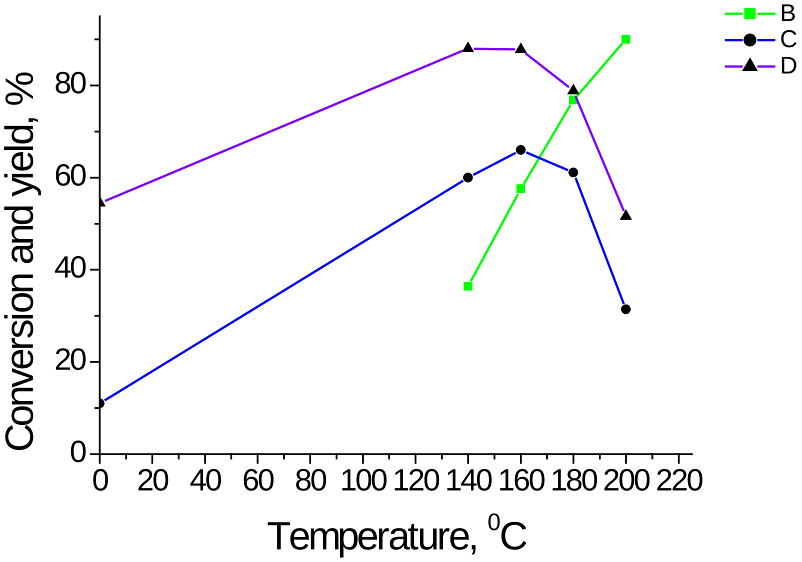

Fig. 2. Conversion of straight-run gasoline on the optimal catalyst. $H_2/CH = 1: 3$; volumetric rate = 2.5 h^{-}.
B - Conversion of $\sum C_{8+}$ %; C - Yield of $\sum(C_5\text{-}C_6)$%; D - Yield of $\sum i\text{-}(C_5\text{-}C_6)$%

Table 1. Influence of the variation of the feed rate of feedstock on the conversion of SRG on the M-11 catalyst at a temperature 180^0C.

WHSV, h^{-1}	1	1.5	2	2.5	3	3.5	4	4.5	5
Conversion of C_{8+}, %	70	67	65	66	64	63	60	57	53
Yield of C_{4-}, %	38	35	33	28	26	20	18	15	10

The volumetric rate control in these experiments was carried out through weighing the amount of catalyst charged into the reactor while maintaining all other reaction parameters. It follows from Table 1 that as the volumetric velocity increases, the conversion of C_{8+} decreases. Moreover, in the range of values 2.0-3.0 h^{-1}, the said conversion practically does not change, and the yield of undesired C_{4-} decreases; the selectivity for target products rises. In general, as can be seen from Table 1, reduced volumetric velocities of 2.0-3.0 h^{-1} are most optimal for low-temperature isomerization treatment of SRG.

The conversion of straight-run gasoline is carried out in hydrogen medium (Fig.3). This effect can be reduced both to the direct effect on the reaction and to the role of the gas carrier which promotes the rapid release of products from the reaction zone.

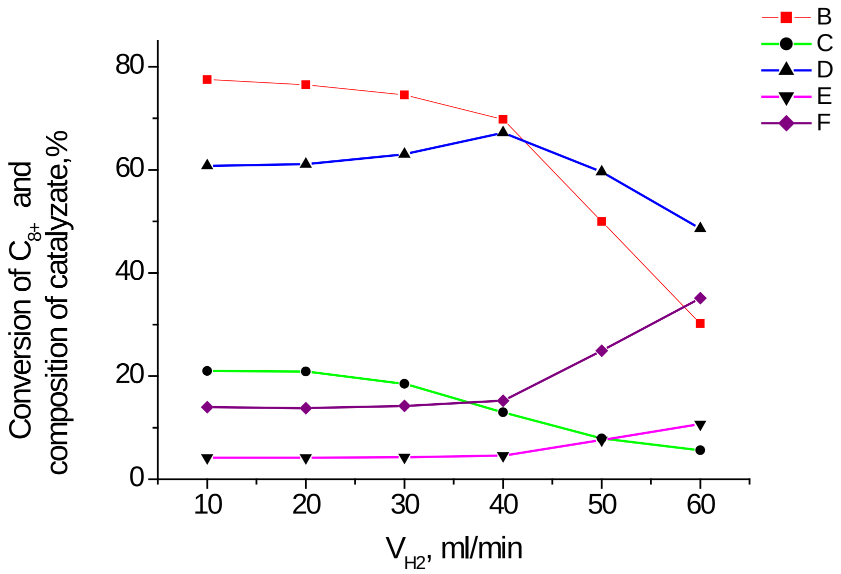

Fig.3. Influence of hydrogen on the SRG conversion. T = 180^0C, volumetric rate = 2.5 h^{-1}; τ = 0.5 h.
B- Conversion of C_{8+}; composition of catalyzate, % (mass): C - C_{4-}; D - C_5-C_6; E - C_7; F - C_{8+}

According to data in Fig.3, there are two areas of hydrogen exposure to the SRG conversion. In the first area when hydrogen is fed at a rate of 10 to 30 ml/min (H_2/CH = 1-3), the process is accompanied by an insignificant change in the C_{8+} conversion when the H_2/CH ratio exceeds 4, the C_{8+} conversion decreases and, simultaneously, increase the concentration of C_7 alkanes. It should be noted that a decrease in the conversion of C_{8+} is accompanied by a similar change in the content of C_{4-} in catalyzate.

In considering that according to [1, 10], the initial stage in the SRG conversion is the primary activation of C_7 and C_{8+} alkanes, it can be assumed that in the H_2/CH ratio range of 1-3, hydrogen is mainly involved in the process of isomerization-disproportionation conversion of SRG.

An increase in the H_2/CH ratio above 4 leads to changes in the effect of hydrogen on the SRG conversion. In the cases above, hydrogen additionally acts as a gas carrier making it difficult to activate the formation of the bimolecular intermediate of the reactant components. The data obtained (Fig.3) suggest that for implementation of the isomerization-disproportionation SRG conversion under atmospheric pressure, the H_2/CH ratio within the range from 2 to 4 is the most optimal, so the ratio H_2/CH = 3 was chosen during the process. Thus, by varying the temperature and volumetric rates, the H_2/CH ratio makes it possible to regulate the low-temperature processing of SRG with minimization of losses of hydrocarbon raw materials in the form of unwanted gaseous alkanes. Thus, as a result of studying the influence of technological parameters of the process on the activity of the catalyst, optimal conditions for conversion of straight-run gasoline on the M-11 catalyst were established.

Table 2 provides data on the accumulation of carbon deposits on M-11 and M-20 catalysts that reveal the effect of temperature reduction and hydrogenolysis metal factor on the process.

Table 2. Accumulation of carbonaceous deposits (CD) on catalysts M-11 and M-20 (M/ HZSM-5/ $SO_4^{2-}ZrO_2$). T = 180^0C, H_2/CH = 3; volumetric rate = 2.5 h^{-1}.

Catalysts	Red. temp., ^OC	Time, hour	CD g	CD %	C(g)	H(g)	C/H
M-11	380	0.5	0.035	0.88	0.03	0.005	0.17
		1.0	0.052	1.31	0.045	0.007	0.16
		2.0	0.056	1.41	0.048	0.008	0.17
	500	0.5	0.018	0.45	0.015	0.003	0.2
		1.0	0.027	0.67	0.023	0.004	0.17
		2.0	0.04	1.0	0.034	0.006	0.18
M-20	380	3.0	0.022	0.55	0.019	0.003	0.16

According to Table 2, the accumulation of carbon compounds on the sample of Co containing M-11with reduction up to 380^0C rises from 0.88% in 15 minutes by 1.6 times in 120 minutes of the experiment to 1.41%. Recovery of M-11 at 500^0C reduces this value to 1%. On the catalyst M-20, by the third hour of operation, the accumulation index of CD makes up 0.55%. Thus, the introduction of nickel reduces the accumulation of carbon deposits.

The table 3 shows the change in conversion C_8 depending on the duration of the experiments on the catalysts under study. Table 3 indicates that during approx. 60 minute of functioning, sample M-11, reduced at 380^0C, begins to lose its activity.

Table 3. Effect of experiment duration on the conversion of C_{8+} on various catalysts.
T = 180^0C, H_2/CH = 3; volumetric rate = 2.5 h^{-1}.

Time, min	Conversion of C_{8+}, %		
	Co, HZSM-5/SO_4^{2-}-ZrO_2, (reduction by hydrogen at 380°C)	Co, HZSM-5/SO_4^{2-}-ZrO_2 (reduction by hydrogen at 500^0C)	Ni, HZSM-5/SO_4^{2-}-ZrO_2 (reduction by hydrogen at 500^0C).
15	76	52	83
60	72	52.5	83.6
120	68	52.8	83.7
180	62	53	84
240	54	54	84.3
300	48	55	84.5

The catalyst M-11 contains 0.4% cobalt; hence, given the low temperature of the catalyst reduction treatment (380^0C), i.e. incomplete reduction of cobalt in the above experiments, the catalyst was further treated with hydrogen at 500^0C. As a result of the treatment, the stability of the catalyst rises; however, the conversion of high molecular weight components is reduced while the composition of the catalyzate remains practically unchanged, the ratio of isocomponents in normal alkanes is maintained. Catalyst M-11, recovered at 500^0 C stably operates within 5 hours.

Thus, at lower temperatures, the elements of group VIII-b play a hydrogenolysing role and get involved in the cleavage process of high-molecular intermediates of reaction. This is confirmed by the mentioned increase in the stability of the C_{8+} conversion after the high-temperature treatment of M-11 in the flow of hydrogen. Since nickel has better recovery ability, the temperature of 380°C is sufficient for its reduction; however, it does not affect SO_4^{2-} anions, so the M-20 catalyst stably operates for, at least, 5 hours with higher activity than the M-11 catalyst. However, the conversion of SRG on M-20 is characterized by a high yield of gaseous alkanes and, especially, methane. Therefore, here Ni- containing catalyst is available as an example to confirm the role of VIII B group elements and achieve the stability of catalytic system functioning and consequently, modification by such elements allows to solve the problem of their stable functioning.

Thus, the rate of deactivation of the used catalysts is related to the rate of accumulation of similar compounds on the surface of the catalyst which is inversely proportional to hydrocracking/hydrogenolysis.

CONCLUSION

1. The influence of technological parameters - temperature (140-200^0C), a volumetric rate (1-$4h^{-1}$) and ratio of hydrogen to the raw material H_2/ SRG on the catalytic properties of 10% ZrO_2, 0.4% Co, 2% SO_4^{2-}/HZSM-5 was examined. Also, optimal process conditions (180^0C, H_2/CH = 3, volumetric rate = 2.5 h^{-1}) were established under which the conversion of C_{8+} is 76.8%, and the sum of C_5-C_6 hydrocarbons, consisting of 78.8% of high-octane isomers, increases from 11.0% to 61.1.2%.
2. The stability of the work of the samples and the amount of carbon compounds formed are determined depending on the duration of the reaction (15-300) min, the temperature of preliminary reduction of the samples by hydrogen (3800C, 5500C) and introduced hydrogenolytic metals (Co, Ni).
3. It revealed that increase in the regeneration temperature of the 10% ZrO_2, 0.4% Co, 2% SO_4^{2-}/HZSM-5 sample from 380 to 500^0C enhances the stability of the catalyst. Replacing Co by Ni more hydrogenolystice increases the stability of the catalyst due to the reduction in the rate of accumulation of carbon deposits; however, in this case the selectivity of the catalyst decreases.

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ВЛИЯНИЕ ТЕХНОЛОГИЧЕСКИХ ПАРАМЕТРОВ НА КАТАЛИТИЧЕСКИЕ И РЕГЕНЕРАЦИОННЫЕ СВОЙСТВА CoHZSM-SO_4^{2-}/ZrO_2 КОМПОЗИЦИОННОЙ СИСТЕМЫ НИЗКОТЕМПЕРАТУРНОЙ ИЗОМЕРИЗАЦИИ ПРЯМОГОННЫХ БЕНЗИНОВ

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Синтезирован композиционый катализатор 10% ZrO_2*, 0.4% Co, 2%* SO_4^{2-}*/HZSM-5 для низкотемпературной конверсии прямогонных бензинов (ПГБ). Исследовано влияние технологических параметров (температуры, объемной скорости и отношения водорода к сырью -* H_2*/ПГБ) на каталитические и регенерационные свойства катализатора. Установлены оптимальные условия процесса (*180^0C*,* $H_2/CH=3$ *; о.с.=2.5* $ч^{-1}$*), при которых конверсия* C_{8+} *составляет 76.8%, а сумма углеводородов* C_5*-*C_6*, состоящая на 78.8% из высокооктановых изомеров, повышается от 11.0 % до 61.1.2%. Изучены регенерационные характеристики катализатора. Стабильность работы образцов и количество образующихся углеродных соединений определены в зависимости от продолжительности реакции, температуры предварительного восстановления образцов водородом и введенных гидрогенолизирующих металлов (Co, Ni). Установлено, что повышение температуры регенерации 10%* ZrO_2*, 0.4% Co, 2%* SO_4^{2-}*/HZSM-5 образца от* 380^0 *до* 500^0C *повышает стабильность катализатора. Замена Co на Ni увеличивает стабильность работы катализатора, благодаря снижению скорости накопления углеродистых отложений, однако при этом селективность катализатора снижается.*

Ключевые слова: *прямогонный бензин, композиционный катализатор, технологические параметры, регенерация, изомеры, углеводород*

BİRBAŞAQOVULMA BENZİNLƏRİNİN AŞAĞI TEMPERATURLU İZOMERLƏŞMƏSİNİN KOMPOZİT CoHZSM-SO_4^{2-}/ZrO_2 SİSTEMİNİN KATALİTİK VƏ REGENERASİYA XASSƏLƏRİNƏ TEXNOLOJİ PARAMETRLƏRİN TƏSİRİ

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Birbaşaqovulma benzinlərinin (BQB) aşağı temperaturlu çevrilməsi prosesi üçün kompozision 10% ZrO_2, 0,4% Co, 2% SO_4^{2-}/HZSM-5 katalizatoru sintez edilmişdir. Texnoloji paramertlərin (temperatur, həcmi sürət və hidrogenin ilkin xammala nisbəti - H_2/BQB) katalizatorun aktivliyinə və regenerasiya xassələrinə təsiri öyrənilmişdir. Prosesin optimal şəraiti (180^0C, H_2/CH = 3 ; h.s.=2,5s^{-1}) təyin edilmişdir. Bu şəraitdə C_{8+}-in konversiyası 76.8% təşkil edir, 78.8%-i yüksəkoktanlı izomerlərdən ibarət olan C_5-C_6 karbohidrogenlərin miqdarı isə 11%-dən 61.2%-ə yüksəlir. Katalizatorun regenerasiya xassələrinin tədqiqi göstərmişdir ki, nümunələrin stabilliyi və əmələ gələn karbon çöküntülərinin miqdarı reaksiyaların davametmə müddətindən, nümunələrin əvvəlcədən hidrogenlə reduksiya edilməsi temperaturundan və daxil edilmiş hidrogenolizləşdirici metaldan (Co, Ni) asılıdır. Müəyyən olunmuşdur ki, 10% ZrO_2, 0.4% Co, 2% SO_4^{2-}/HZSM-5 nümunəsinin regenerasiya temperaturunun 380-dən 500^0C-yə yüksəldilməsi katalizatorun stabilliyini artırır. Co-ın Ni ilə əvəz edilməsi katalizatorun stabilliyini karbon çöküntülərinin toplanma sürətinin azalması hesabına artırır, lakin bu zaman katalizatorun selektivliyi aşağı düşür.

Açar sözlər: *birbaşaqovulma benzinləri, kompozision katalizatoru, texnoloji paramertlər, regenerasiya, karbohidrogenlər.*