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SELECTIVE HYDROGENATION OF ISOPRENE, PIPERYLENE AND THEIR MIXTURES ON SKELETAL NICKEL CATALYSTS

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Abstract: Results of the hydrogenation process of isoprene and piperylene mixtures, as well as their mixtures on multicomponent skeletal nickel catalysts are reported in this article. Hydrogenation of piperylene and isoprene proceeds at a higher rate ($W=215-220 \text{ cm}^3/\text{min}\cdot\text{g Ni}$) on Ni-Mo-Cu alloy catalyst than on skeletal nickel ($W=112-115 \text{ cm}^3/\text{min}\cdot\text{g Ni}$). The results of chromatographic analysis indicate that the hydrogenation of piperylene proceeds with high selectivity. The selectivity coefficient K_s is 0.95 and 0.98 on skeletal nickel and Ni-Mo-Cu catalyst, respectively. At hydrogenation of the piperylene-isoprene mixture the piperylene conversion rate ($P > J$) is prevailed. At piperylene conversion degree $P=50 \%$ the isoprene J conversion is 35-37 % on Renea Nickel. 34 % on Ni-Mo-Cu catalyst. The selectivity degree of hydrogenation of this mixture is $S_{p-i} = 0.34-0.36$ on

дәрежесінде Р=50% изопренниң конверсиясы Реней никелінде 35-37% құрайды, ал Ni-Mo-Cu катализаторында 34 %. Бұл қоспаның гидрленуінің селективтілік дәрежесі Реней никелінде $S_{\text{п-и}} = 0,34-0,36$, ал Ni-Mo-Cu катализаторында $S_{\text{п-и}} = 0,40$. Пиперилен – изопрен қоспаларын Ni-Al-Mo-Cu (42-50-3-5 %) катализаторлар қатысында гидрлеу кезінде моно-және алмастырылмаган ацетилендер изопренмен салыстырғанда үлкен трансформация дәрежесімен қанықтырылатыны анықталды.

Түйін сөздер: талғампазды гидрлеу, изопрен, пиперилен, қаңқалы никель катализаторлары

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СЕЛЕКТИВНОЕ ГИДРИРОВАНИЕ ИЗОПРЕНА, ПИПЕРИЛЕНА И ИХ СМЕСЕЙ НА СКЕЛЕТНЫХ НИКЕЛЕВЫХ КАТАЛИЗАТОРАХ

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Аннотация: В настоящей статье приведены результаты процесса гидрирования смесей изопрена, пиперилена и их смесей на многокомпонентных скелетных никелевых катализаторах. На катализаторе из сплава Ni-Al-Mo-Cu гидрирование пиперилена и изопрена идет с большой скоростью ($W=215-220 \text{ см}^3/\text{мин} \cdot \text{г Ni}$), в отличие от скелетного никеля ($W=112-115$

см³/мин·г Ni). Результаты хроматографического анализа свидетельствуют, что гидрирование пиперилена протекает с высокой селективностью. Коэффициент селективности K_s составляет 0,95 и 0,98 на скелетном никеле и Ni-Mo-Cu катализаторе, соответственно. При гидрировании смеси пиперилен-изопрен преобладает скорость превращения пиперилена ($P > J$). При степени превращения пиперилена $P=50\%$ конверсия изопрена J составляет 35-37 % на Nial. 34 % - на Ni-Mo-Cu катализаторе. Степень селективности гидрирования этой смеси $S_{п-и} = 0,34-0,36$ на Nial, $S_{п-и} = 0,40$ на Ni-Al-Mo-Cu. Установлено, что при гидрировании смесей пиперилен – изопрен на Ni-Al-Mo-Cu (42-50-3-5 %) катализаторах моно- и дизамещенные ацетилены насыщаются с большой степенью превращения по сравнению с изопреном.

Ключевые слова: селективное гидрирование, изопрен, пиперилен, скелетные никелевые катализаторы.

Introduction

Hydrogenation of unsaturated hydrocarbons is an industrially important process underlying the production of fuels that meet modern requirements for their quality, polymers, synthetic rubbers. Modernization of existing, development of new effective, environmentally friendly (Shafigulin et al., 2017; Mikhailova et al, 2014), economically more profitable technologies is a priority direction of modern science and economics (Khorkova et al, 2010; Konkova et al, 2013). An important point here is the development of inexpensive catalysts based on new materials that provide an acceptable value of catalytic activity comparable to that shown by the catalysts currently used in industry (Lakshmi et al, 2016).

The most applicable for this purpose are nickel-based hydrogenation catalysts, which have high activity and relatively low cost compared to catalysts based on noble metals (Kasyanova et al, 2016; Putilin et al, 2015; Statsenko et al, 2016; Kairbekov et al, 2023).

In industry, nickel catalysts are mainly used for hydrogenation processes. This is due to the high activity and selectivity, ease of preparation and regeneration, stability of operation in a long cycle, resistance to poisoning by catalytic poisons (Karakhanov et al, 2014; Boymans, 2015). It has been shown that the modification of skeletal nickel by various metals makes it possible to regulate the properties of the catalyst in a wide range (Jeldybaeva et al, 2022; Jeldybayeva et al, 2021).

Thus, the search for new cheap, highly active and selective catalysts for hydrogenation of individual unsaturated hydrocarbons and their technical mixtures is a task of great practical importance.

In this paper, studies were carried out on the selective hydrogenation of isoprene, piperylene and their mixtures on skeletal nickel catalysts made of Ni-al and Ni-Al-Mo-Cu alloys.

Materials and methods

Raw materials, catalysts, experimental methods

Table 1 shows the physicochemical characteristics of piperylene and isoprene.

Table 1: Main physicochemical characteristics of piperylene and isoprene

Connection	The structural formula	Molecular weight	Boiling point, °C	Density d_4^{20}	Refractive index n_D^{20}	Solubility in:		
						water	ethanol	hexane
Isoprene (2-methyl-1-3-butadiene)	$\text{CH}_2=\text{C}-\text{CH}=\text{CH}_2$ CH ₃	68.12	34.07	0.6806	1.4194	not soluble	∞	∞
Piperylene (1,3-pentadiene)	$\text{CH}_2=\text{CH}-\text{CH}=\text{CH}-\text{CH}_3$	68.12	44.07 (c) 42.03 (t)	0.6910 0.6760	1.3883	not soluble	∞	∞

Preparation of catalysts. The weighed amount (0.4-0.8 g) of the ground, powdered Ni-Al-alloy from the fractions of 0.06-0.20 mm was treated with a 20 % KOH solution at a temperature of 96 °C in a boiling water bath for 2 hours. The obtained products were washed from the alkali by decantation with distilled water 4-5 times, until a negative reaction to OH-ions in washing water was obtained. The catalyst was then washed with a solvent in which the hydrogenation process was carried out (Jeldybayeva et al, 2021).

Methodology of the study. Hydrogenation was carried out in a thermostatically controlled catalytic “chamber” at atmospheric pressure and a temperature of 20 °C. Simultaneously, the reaction rates (the amount of absorbed hydrogen per unit time, cm³/min) and the catalyst potential (mV) relative to the calomel reference electrode were recorded according to the methodology (Kairbekov et al, 2021). Before the reaction, the catalyst was treated with hydrogen in a solvent ($V = 25 \text{ cm}^3$) until a reversible hydrogen potential was established. Hydrogenation was carried out in the kinetic mode (700-800 pumps/min).

The selectivity index (α) was determined graphically using equation (1). The selectivity index is determined by both kinetic and thermodynamic factors, together reflecting the presence of energy and structural correspondence:

$$\alpha = \frac{K_2 \times B_2}{K_1 \times B_1} \quad (1)$$

where K_1, K_2 are the hydrogenation rate constants of the 1st and 2nd substances; B_1, B_2 are the adsorption coefficients of the hydrogenated substances on the hydrocarbon surface.

The degree of selectivity (S) of hydrogenation of the mixture of substances was calculated by formula (2):

$$S = 1 - \alpha \quad (2)$$

The ratio of adsorption coefficients B_2/B_1 was determined using equation (3):

$$\frac{W_2}{W_1} = \frac{K_2 B_2 C_2}{K_1 B_1 C_1} \quad (3)$$

where K_1, K_2 are the rate constants of the hydrogenation reaction of 1,2 substances, B_1, B_2 are the adsorption coefficients of the components of the mixture, C_1, C_2 are the concentrations of the components in the initial mixture.

The selectivity coefficient, migration coefficient and hydrogenation isomerization coefficient of isoprene and piperylene were determined by the following formulas (4-6).

$$K_s = \frac{\text{alkene output, \%}}{\text{output (alkene+alkane) \%}} \quad (4)$$

$$K_{migr} = \frac{\text{output (cis-alkene-2+trans-alkene-2)}}{\text{output (alkene-1+cis-alkene-2+trans-alkene-2)}} \quad (5)$$

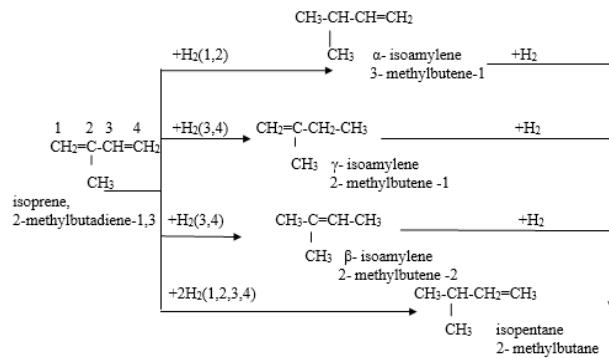
$$K_{ism} = \frac{\text{output trans-alkene-2, \%}}{\text{output (cis-alkene-2+trans-alkene-2), \%}} \quad (6)$$

Chromatographic analysis was performed on a Chromos GC-1000 chromatograph (“Chromos”, Russia) with a flame ionization detector in isothermal mode using a BP21 capillary column (FFAP) with a polar phase (PEG modified with nitroterephthalate) 50 m long and 0.32 mm inner diameter. The column was maintained at a temperature of 90 °C, the temperature in the evaporation chamber was 200 °C, the carrier gas was helium, the volume of the injected sample was 0.2 µl. During the experiment, 2-3 samples of the liquid reaction mixture were taken for analysis (Jeldybayeva et al, 2021).

Results and discussion

Isoprene hydrogenation

To clarify in detail, the mechanism of hydrogenation of mixtures of hydrocarbons, the hydrogenation of individual components was previously studied. The connection of hydrogen to the conjugated system of isoprene bonds on the studied catalysts goes in all possible directions (scheme 1):



Scheme-1

The rate of hydrogenation of isoprene to the absorption of about half of the calculated amount of hydrogen remains constant. At the moment of attachment of one mole of hydrogen, a sharp fracture is observed ("critical point of the conjugated system"), and then the intermediate isoamlyenes are saturated at a significantly slower rate (1.5-2 orders of magnitude) than the diene bond (Fig. 1).

Modification of the skeletal nickel Mo-Cu leads to a decrease in the adsorption capacity of the catalyst for isoprene ($\Delta E = 190$ mV) and an increase in selectivity from 0.96 to 0.98 (Table 2).

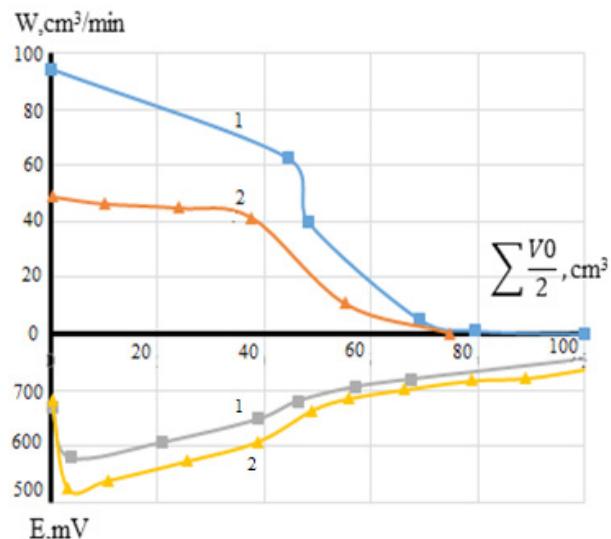


Figure 1: Kinetic and potentiometric curves of isoprene hydrogenation ($A_{h_2}=100 \text{ cm}^3\text{H}_2$)
In ethanol on skeletal nickel catalysts made of alloys:
1-Ni-al (50-50 %), 2-Ni-Al-Mo-Cu (45-50-5) ($m=0.8 \text{ g}$)

1:1	Ni-Al	117	260	35	90	0.64	1.6	0.36	0.98	0.98
1:3	Ni-Al	120	240	37	93	0.66	4.5	0.34	0.98	0.98
1:1	Ni-Al-Mo-Cu	218	220	34	87	0.60	1.7	0.40	0.99	0.99

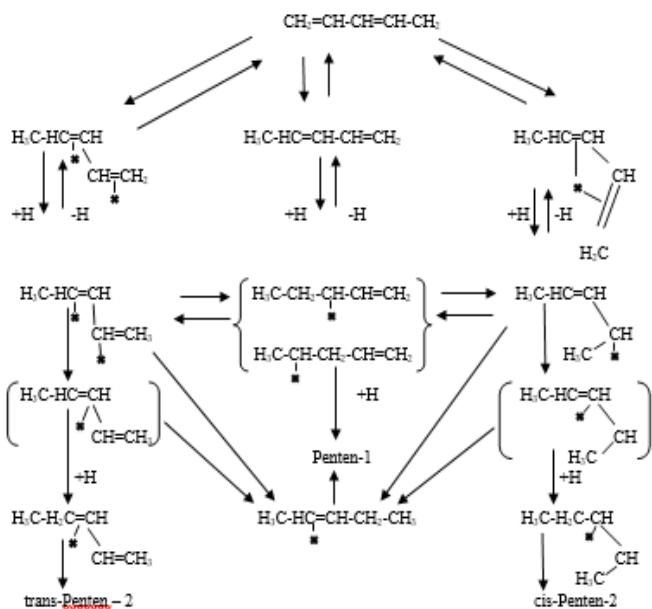
P, J – degree of conversion of piperylene, isoprene (relative %) at absorption of 50 cm³ of H₂

Hydrogenation of piperylene

For hydrogenation reactions, piperylene with a ratio of trans/cis-stereoisomers equal to 2 was used (Table 2, Fig. 3.4). Fracture on the kinetic curve of hydrogenation of piperylene ($A_{H_2} = 100 \text{ cm}^3$) corresponds to the addition of 1 mole of hydrogen to piperylene (Fig. 3). The absorption of 2 moles of hydrogen proceeds at a significantly lower rate (by 1-2 orders of magnitude).

On a Ni-Al-Mo-Cu alloy catalyst, piperylene is hydrogenated at a higher rate ($W = 215 \text{ cm}^3/\text{min} \cdot \text{g Ni}$) than on skeletal nickel ($W = 112 \text{ cm}^3/\text{min} \cdot \text{g Ni}$). The results of chromatographic analysis indicate that the hydrogenation of piperylene proceeds with high selectivity. The Selectivity factor is 0.95 and 0.98 on skeletal nickel and Ni-Mo-Cu catalyst, respectively (Kairbekov et al, 2023).

When piperylene is hydrogenated on skeletal nickel catalysts (Table 2, Fig. 4), pentene-1, cis- and trans-pentenes-2 are formed, and in insignificant amounts - pentane (Selectivity factor = 0.95 on Nial, Selectivity factor = 0.98 on Ni-Al-Mo-Cu). The formation of pentene isomers is shown in Scheme 2 below.



Scheme-2

The ratio of the rates of formation of pentene-1; trans-pentene-2; cis-pentene-2 is approximately constant and is 2:2:1 (Fig. 4). After piperylene disappears from the reaction mixture, pentene-1 is hydrogenated at a higher rate. At the same time, the content of trans-pentene-2 and cis-pentene-2 increases (mainly the trans isomer), which indicates the isomerization of pentene-1.

When hydrogenating alkadienes (piperylene, isoprene), high selectivity (Selectivity factor of isoprene), constancy of the ratios of the amounts formed by C₅-alkenes, as well as their lack of isomerization at the first stage are associated with adsorption displacement of alkenes by alkadienes on the surface of catalysts.

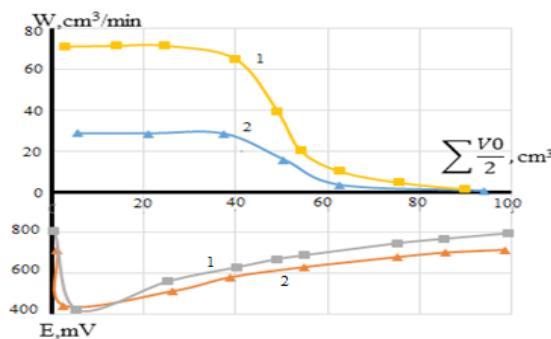
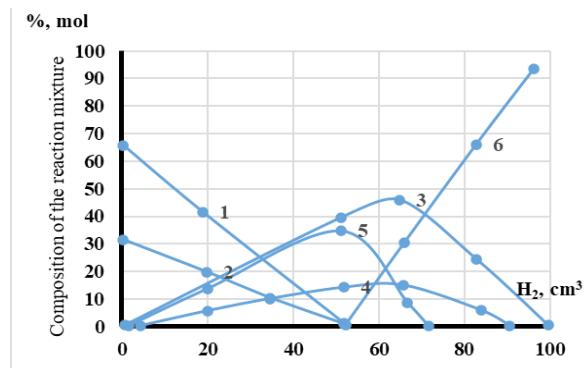


Figure 3: Kinetic and potentiometric hydrogenation curves of piperylene ($A_{H_2} = 100 \text{ cm}^3$) in ethanol on skeletal nickel alloy catalysts:
1 - Ni-Al-Mo-Cu (42-50-3-5%), 2 - Ni-al (50-50%) ($m = 0.8 \text{ g}$)



1 - trans-piperylene, 2 - cis-piperylene, 3 - trans-pentene-2, 4 - cis-pentene-2,
5 - pentene-1, 6 - pentane.

Figure 4: Diagram of the composition of the hydrogenation catalyst of piperylene on a skeletal catalyst from the Ni-Al-Mo-Cu alloy (42-50-3-5 %)

Hydrogenation of mixtures of piperylene with isoprene

To assess the possibility of joint removal of piperylene impurities from isoprene, along with monosubstituted acetylenes, by selective catalytic hydrogenation, the hydrogenation processes of the following mixtures were studied: piperylene-isoprene (100:100 cm³ H₂), on catalysts made of Ni-al (50-50 %), Ni-Al-Mo-Cu (42-50-3-5 %) alloys in ethanol at a temperature of 20 °C (Table 2, Fig. 5).

The results of the hydrogenation curves (Fig. 5) and chromatographic studies (Fig. 6) of the mixture of piperylene-isoprene on skeletal nickel catalysts from Ni-al and Ni-Al-Mo-Cu alloys in ethanol showed that the hydrogenation+ rate of the mixture before absorbing about half of the calculated amount of hydrogen remains constant, and then the fracture and saturation of the formed piperylenes and isoamylanes is observed at a much lower rate.

When hydrogenating the piperylene-isoprene mixture on skeletal nickel catalysts, the conversion rate of piperylene ($P > J$) predominates. At the degree of conversion of piperylene P=50 %, the conversion of isoprene J is 35-37 % on Raney nickel, 34 % on Ni-Mo-Cu catalyst. The degree of hydrogenation selectivity of this mixture is = 0.34-0.36 on Raney nickel, and 0.40 on Ni-Mo-Cu.

The saturation of the piperylene-isoprene mixture is characterized by the preferential addition of hydrogen to the piperylene. Piperylene is saturated more intensively than isoprene. The degree of conversion of hydrocarbons at the time of absorption of 50 cm³ of H₂ on a Ni-Mo-Cu catalyst is 36 and 22 % for piperylene and isoprene, respectively.

The alkadien mixtures used are hydrogenated at a higher rate on a Ni-Mo-Cu catalyst ($W = 218 \text{ cm}^3/\text{min} \cdot \text{g Ni}$) than on Raney nickel ($W = 117-120 \text{ cm}^3/\text{min} \cdot \text{g Ni}$). The addition of the Mo-Cu composition reduces the adsorption of alkadienes on the catalyst surface ($\Delta E = 240-260 \text{ mV}$ on Raney Nickel, $\Delta E = 220 \text{ mV}$ on Ni-Al-Mo-Cu).

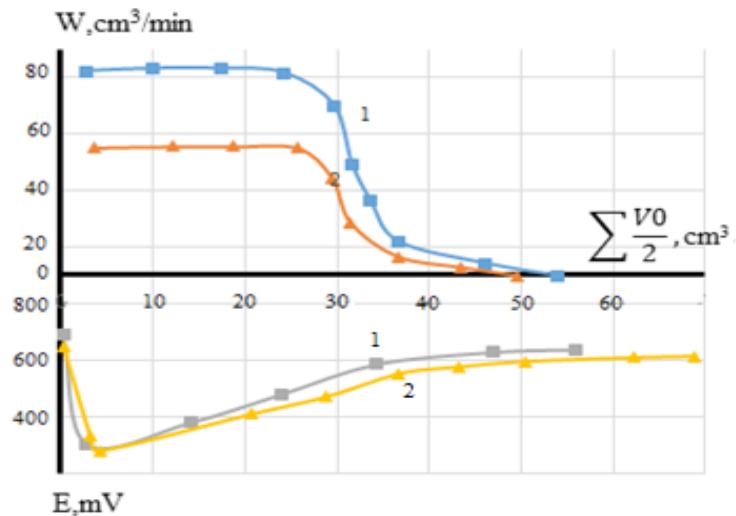
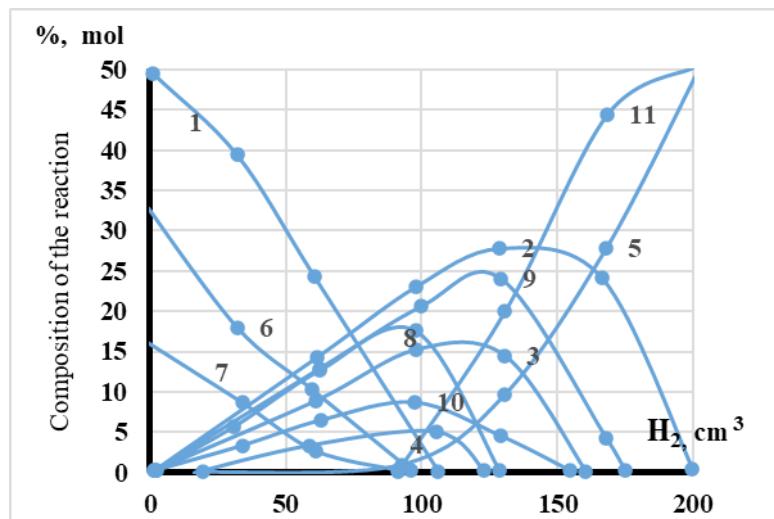


Figure 5: Kinetic and potentiometric hydrogenation curves
1.3 – mixtures of isoprene – piperylene (100:100 $\text{cm}^3 \text{H}_2$) in ethanol on skeletal nickel catalysts from alloys:
1 – Ni-Al-Mo-Cu (42-50-3-5 %), 2 – Ni-al (50-50 %) ($m = 0.8 \text{ g}$)



1-isoprene, 2- β -isoamylene, 3- γ -isoamylene, 4- α -isoamylene, 5-isopentane,
6 – trans-piperylene, 7 – cis-piperylene, 8 – pentane-1, 9 – trans-pentene-2,
10 – cis-pentene-2, 11 – pentane.

Figure 6: Diagrams of the composition of the hydrogenation catalyst of piperylene and isoprene on a skeletal catalyst from the Ni-Al-Mo-Cu alloy (42-50-3-5 %)

The introduction of small Mo additives to the Ni-al alloy in combination with Cu, slightly reducing the strong promotional effect of Cu, contributes to the formation of a selective and stable catalyst. A favorable combination of Mo and Cu is associated, apparently, with the presence of the following factors: the introduction of Mo promotes the strengthening of the Me-H bond due to the formation of Mo oxides, while Cu additives lead to an increase in the adsorption capacity of the -C=C- bond of alkanes. Modification of Mo complicates the phase composition of the catalyst both due to partial oxidation of Ni and the formation of significant amounts of Al_2O_3 . When Mo and Cu are co-administered, the stabilizing effect of the formed metal oxides is enhanced, which prevents the catalyst from recrystallization and reduces activity.

Conclusion

On a Ni-Al-Mo-Cu alloy catalyst, piperylene and isoprene are hydrogenated at a higher rate ($W = 215\text{-}220 \text{ cm}^3/\text{min}\cdot\text{g Ni}$) than on skeletal nickel ($W = 112\text{-}115 \text{ cm}^3/\text{min}\cdot\text{g Ni}$).

According to the chromatographic analysis of the catalyst composition during the hydrogenation reaction of isoprene, it follows that in the first stage - before the absorption of approximately 1 mole of hydrogen - isoamylene is predominantly formed on the studied catalysts (Selectivity factor of isoprene = 0.96-0.98), and isopentane are formed only in insignificant amounts (1-2%).

Hydrogenation of piperylene also proceeds with high selectivity. The selectivity factor is 0.95 and 0.98 on skeletal nickel and Ni-Mo-Cu catalyst, respectively.

The hydrogenation of the piperylene-isoprene mixture is dominated by the conversion rate of piperylene ($P > J$). At the degree of conversion of piperylene $P=50\%$, the conversion of isoprene J is 35-37 % on Raney nickel, 34 % on Ni-Mo-Cu catalyst. The degree of hydrogenation selectivity of this mixture is 0.34-0.36 on Raney nickel, and 0.40 on Ni-Al-Mo-Cu.

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