

3-27-2021

RESEARCH OF THE PROCESS OF CONTINUOUS HYDROGENATION OF BENZENE IN THE PRESENCE OF MODIFIED CATALYSTS

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Recommended Citation

Kedelbaev, B Dr. Professor; Lakhanova, K PhD; Iztleuov, G; and Turabdzhanov, Sadritdin Dr., Professor (2021) "RESEARCH OF THE PROCESS OF CONTINUOUS HYDROGENATION OF BENZENE IN THE PRESENCE OF MODIFIED CATALYSTS," *Technical science and innovation*: Vol. 2021: Iss. 1, Article 1.

DOI: <https://doi.org/10.51346/tstu-01.21.1-77-0099>

Available at: <https://btstu.researchcommons.org/journal/vol2021/iss1/1>

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UDC 541.128

RESEARCH FOR THE CONTINUOUS BENZENE HYDROGENATION PROCESS IN THE PRESENCE OF THE MODIFIED CATALYSTS

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Abstract. *The technology has been developed for the production of unsaturated alloy nickel hydrogenation catalysts. The results of the study of the phase, chemical compositions and structure of nickel alloys and catalysts showed that the ferroalloy introduction as a modifying additive affects the NiAl₃ / Ni₂Al₃ ratio. The studied alloying metals are practically insoluble in*

alkali and exist in the catalyst in dissolved states. All these changes favorably affect the catalytic properties of modified nickel catalysts in the benzene hydrogenation reaction. A systematic study of the stationary catalyst activity with addition of ferroalloys in the continuous catalytic benzene hydrogenation was carried out with a wide variation of the process parameters. All catalysts are dominated by particles with $R = 0-2$ microns, the concentration of which reaches 75-89%. Table 1 shows that, with an increase in the amount of additives in alloys from 3 to 9% by weight, the concentration of particles with $R = 0-2 \mu\text{m}$ in catalysts decreases differently within 89-75%, depending on the nature of the alloying metals. In addition, modifying additives also increase the concentration of particles with $R = 2-4 \mu\text{m}$. The results of optical microscopy show that almost all studied skeletal nickel catalysts are enriched by 90-99% in particles with $R_{\text{max}} = 1-5 \mu\text{m}$. A systematic study of the stationary catalyst activity with addition of ferroalloys in the reaction of continuous catalytic benzene hydrogenation with a wide parameter variation of the technological process has been carried out. At the same time, it is highly active, stable and selective for cyclohexane new stationary catalysts for industrial purposes operating at temperatures up to 140°C and pressure up to 8 MPa.

Keywords: *catalyst, nickel, aluminum, benzene, cyclohexane, cadmium plating, promotion, ferroalloys*

INTRODUCTION. The petrochemical potential of industrialized countries is determined by the volume of production and processing of aromatic hydrocarbons. It is these products that form the raw material base of the organic synthesis industry [1]. At present, the general trend in the development of the oil industry in the world is the tightening of environmental legislation aimed at reducing harmful emissions when using fuels and oils, as well as a constant increase in requirements for the quality of petroleum products.

The tightening of requirements and standards for quality leads to the need for advanced development of technologies for their production. Research into the hydrogenation of aromatic hydrocarbons is one of the most important problems in petrochemistry due to the tightening of requirements for modern motor fuels, recently in accordance with the requirements of Euro-5 (2)

The problem of the aromatic hydrocarbon content reduction can be solved by many methods, including their extraction by adsorption or extraction, as well as conversion into other, less harmful compounds. One of the most effective methods for improving the operational properties of fuels, in particular gasoline, is their hydrodearomatization, which consists in the process of hydrogenation of aromatic hydrocarbons in the presence of catalysts.

From this point of view, the solution of the problems of dearomatization of such compounds is an urgent problem.

A review of the literature data on the hydrogenation of benzene [3-7] shows that in many cases the process is carried out in the vapor phase at temperatures of $250-325^\circ\text{C}$ and a hydrogen pressure of 10.0-27.0 MPa. Platinoids are also used as catalysts. Naturally, under these conditions, the products of isomerization and cleavage are observed in the catalyst, which reduces the quality of the target product, cyclohexane.

New methods have been discovered to increase the activity, stability and mechanical strength of nickel catalysts by introducing additives of other metals into the alloy at the time of

their preparation [8-10]. As a result, effective catalysts have been found that accelerate the benzene hydrogenation reaction. The hydrogenation of aromatic hydrocarbons was carried out on them in a reactor periodic action in the temperature range 20 - 200 ° C and hydrogen pressure 0.1-15.0 MPa.

On an industrial scale, in the production of cyclohexane, coprecipitated oxide copper-chromite catalysts are widely used [11-13], although they are not devoid of significant disadvantages in terms of catalytic and operational properties in hydrogenation processes. In connection with the above, researchers have proposed numerous modified Ni, Cu, Co -alloy catalysts [14-15], which have not yet acquired industrial application in these industries. Therefore, it is urgent to improve these catalysts by modifying them in order to further increase their selectivity, operational and catalytic properties in the processes of hydrogenation of aromatic hydrocarbons in the liquid phase.

In works (16-17) it is shown that alloyed nickel catalysts modified with ferroalloys are the cheapest and most accessible in hydrogenation processes.

This article presents the results of a study of the hydrogenation of benzene to cyclohexane on alloyed nickel catalysts modified with ferroalloys (FS) - ferromolybdenum (FMo), ferrosilicochromium (FSKh) and ferrosilicocalcium (FSK).

EXPERIMENTAL PART. The initial alloys were prepared in a high-frequency furnace, the content of the components in them varied (in wt%): nickel - 45-49, aluminum - 50, FS - 1.0-5.0. The original alloys were used in the form of various fractions, depending on the method of their study.. So, Alloys with different diameters were used for metallographic and local X-ray spectral analysis. So for continuous hydrogenation used alloys with a grain diameter and 3-5 mm. Physicochemical studies were carried out in the presence of powdered alloys and catalysts. For this, the starting alloys were crushed and sieved through a sieve 0.25 mm. The catalysts were prepared by leaching the alloys with a 20% aqueous solution NaOH in a boiling water bath for 1 hour, then washed with water from alkali to neutral reaction with phenolphthalein. The catalyst was activated directly in the reactor itself. In this case, a granular alloy with linear dimensions of 2-4 mm (100 g) was placed in a special glass attached to the bottom of the reactor and the leaching process was carried out with a 10% aqueous solution of sodium hydroxide.

In the course of leaching with the help of a metering pump, the reactor was first filled with distilled water, and then an alkali solution was fed from the "measuring tank". The degree of aluminum removal was monitored by the evolution of hydrogen, the amount of which was changed with a gas meter. After the release of the required amount of hydrogen to the leached catalyst, using the same pump, to stop the leaching process and wash the catalyst from alkali residues to pH-7, water was supplied from the "measuring tank". The amount of aluminum removed from the consistency was about 10-15%.

To identify the obtained alloys and catalysts, studies of their structure and adsorption properties were carried out. For this purpose, the catalysts were prepared by leaching the alloys with a 20% aqueous solution of sodium hydroxide in a boiling water bath for 1 hour. Samples taken during the hydrogenation studies were analyzed by chromatographic and partially refractometric methods with the construction of calibration curves relative to benzene, cyclohexane and

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cyclohexene. The sensitivity of the detector of the chromatograph in a flow of helium to the assumed composition of the catalyzate, which is formed during the hydrogenation of benzene according to the Porter Formula $A = S \cdot V \cdot \ln \frac{1}{q} / \nu$, where S is the peak area, cm²; V-velocity of the gas-carrier ml / min., V1-recorder sensitivity mv / s; ν - speed of the chart tape cm / min., q - quantitative value of the sample, mg. The calibration coefficients were for cyclohexane $K_{c6n6} = 0.974$, $K_{c6n10} = 0.972$, for cyclohexane $K_{c6n12} = 1.057$. The detector sensitivity was $A_{c6n6} = 3.5 \cdot 10^{-2}\%$; by $A_{c6n10} = 4.6 \cdot 10^{-2}\%$; by $A_{c6n12} = 6.8 \cdot 10^{-2}\%$; and for propane = $2 \cdot 10^{-2}\%$. The found values of the detector sensitivity are, respectively, for benzene- $3.5 \cdot 10^{-2}\%$, for cyclohexane - $6.8 \cdot 10^{-2}\%$, based on cyclohexene- $4.6 \cdot 10^{-2}\%$. Technical characteristics of the sensitivity of the device relative to propane- $2 \cdot 10^{-2}\%$. The catalytic activity of the studied catalysts was compared mainly by chromatography data.

RESULTS AND DISCUSSION. Since nickel catalysts have been studied sufficiently[nine], we limited ourselves to presenting data on phase composition, structure; specific surface area of alloys and catalysts based on aluminum-nickel alloys modified with ferroalloys.

It should be noted that in the literature, the influence of ferroalloys on the physicochemical properties of alloyed aluminum-nickel catalysts is insufficiently illuminated. In this regard, we have investigated the effect of FSC, FMo, and FSH on the phase composition and structure of aluminum-nickel alloys and catalysts.

From table 1, it can be seen that modifying additives have a significant effect on the qualitative and quantitative composition and structure of the starting alloys and catalysts. The catalysts are composed of skeletal nickel- Al_2O_3 , Ni_2Al_3 and ferroalloy. Modifying additives do not affect the crystal lattice parameter of nickel, but they significantly grind its crystals, and also increase the specific surface area of the catalyst before $130.0 \text{ m}^2 / \text{g}$.

Table 1.

Characteristics of aluminum-nickel alloys and catalysts with addition of ferroalloys

| Modifying additives | Alloys | | | | | Catalysts | | |
|--------------------------|-------------------|---------------------------------|---------------------------------|----|-------------------|-----------------------------------|----------------------|--|
| | phase area, % | | | Fx | NiAl ₃ | Crystal lattice parameter (a), nm | Crystal size (L), nm | Specific surface area (S) m ² / g |
| | NiAl ₃ | Ni ₂ Al ₃ | Al + NiAl ₃ eutectic | | | | | |
| Ni - Al = 50 - 50 | | | | | | | | |
| - | 50y | 40 | 10 | - | 1.25 | 0.353 | 5.4 | 1.5 |
| Ni - 50% Al - Φ CTO | | | | | | | | |
| 3-10.0 | 50 | 39 | 7 | 3 | 1.28 | 0.353 | 4.7 | 110 |
| Ni - 50% Al - Φ Mo | | | | | | | | |
| 3-10.0 | 48 | 44 | 12 | 6 | 1.33 | 0.353 | 4.6 | 130 |
| Ni - 50% Al - FSH | | | | | | | | |
| 3-10.0 | 44 | 39 | 12 | 8 | 1.33 | 0.353 | 3.2 | 122.4 |

Thus, the introduction of ferroalloy additives into an aluminum-nickel alloy significantly affects the phase composition, structure, and specific surface area of skeletal nickel catalysts. The results of X-ray and X-ray spectral studies have shown that the modifying metals in the catalysts are not in a free, but in a dissolved state. The dispersion of catalysts affects the activity and selectivity of catalysts, and the main reason for the change in activity is the dispersion of catalyst grains and an increase in the efficiency factor, which simulates chemical promotion.

The particle size distribution of skeletal nickel catalysts with additions of FSKh, FSK and FMo ferroalloys has been investigated. The data of microscopic and electron microscopic studies of the particle size distribution of skeletal nickel catalysts are shown in Table 1.

All catalysts are dominated by particles with $R = 0-2$ microns, the concentration of which reaches 75-89%. Table 1 shows that, with an increase in the amount of additives in alloys from 3 to 9% by weight, the concentration of particles with $R = 0-2 \mu\text{m}$ in catalysts decreases differently within 89-75%, depending on the nature of the alloying metals. In addition, modifying additives also increase the concentration of particles with $R = 2-4 \mu\text{m}$. The results of optical microscopy show that almost all of the studied skeletal nickel catalysts are enriched by 90-99% in particles with $R_{\text{max}} = 1-5 \mu\text{m}$.

Table 2.

Results of microscopic and electron microscopic studies of skeletal nickel catalysts.

| Catalyst | Particle size distribution% by size R, μm | | | | | |
|--------------|--|-----|-----|-----|-----|------|
| | 0-2 | 2-4 | 4-6 | 6-8 | > 8 | T3 |
| Ni (50% Al) | 77 | 8 | 6 | 2 | 7 | 0.12 |
| Ni-3-10% FSK | 78 | 8 | 8 | 4 | 2 | 0.45 |
| Ni-3-10% FMo | 85 | 6 | 5 | 2 | 1 | 0.35 |
| Ni-3-10% FSH | 83 | 6 | 6 | 3 | 1 | 0.36 |

The results of studying the particle size distribution using optical microscopy and an electron microscope confirm the enrichment of skeletal nickel catalysts with particles with $R_{\text{max}} = 1 \mu\text{m}$. Modifying metals increase the proportion of particles with a size of 0-6 microns.

We have studied [2] the porous structure of skeletal nickel catalysts with addition of ferroalloys. Argon sorption isotherms show that the forms of hysteresis loops for most of the modified nickel catalysts are characterized by a parallel arrangement of adsorption and desorption branches in the middle range of relative pressures and, according to the de Boer classification, belong to type A, which indicates the prevalence of cylindrical pores [3].

Table 3. the parameters of the porous structure of skeletal nickel (50% Al) catalysts with addition of ferroalloys are given. It is seen that modifying ferroalloys mainly increase SBET, SKUM, respectively, to 110-130.5 and 85-98 m^2 / g ; pore volume - 1.1-1.4 times; effective pore radius REFF - 1.06-1.5 times. A simultaneous increase in the specific surface area and pore volume with relatively high effective radius apparently due to the dispersion of the nickel phase of the catalysts with modifying metals.

Table 3.

Parameters of the porous structure of skeletal aluminum-nickel catalysts with addition of ferroalloys.

| Catalyst | SBET, m^2 / r | SCUM, m^2 / r | $\frac{S_{\text{БЭТ}} - S_{\text{КУМ}}}{S_{\text{БЭТ}}}$ | Vthr, cm^3 / r | Reff, Å | Isother m type |
|----------|----------------------------------|----------------------------------|--|-----------------------------------|---------|-------------------|
|----------|----------------------------------|----------------------------------|--|-----------------------------------|---------|-------------------|

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|----------------|-------|----|------|-------|----|---|
| | | | 100% | | | |
| Ni (50% Al) | 105 | 75 | 28.5 | 0.105 | 30 | A |
| Ni - 3-10% FSK | 110 | 85 | 22.7 | 0.120 | 34 | A |
| Ni - 3-10% ΦMo | 130.5 | 98 | 24.9 | 0.138 | 36 | A |
| Ni - 3-10% FCX | 123.7 | 92 | 23.9 | 0.148 | 36 | A |

The results of studying the kinetics of desorption of H₂ from skeletal nickel catalysts with addition of ferroalloys are given in Table 3. It can be seen that the desorption of hydrogen starts from 0°C and occurs continuously up to 6000°C and above, but at an unequal rate; maximum in the areas 100-1400°C and 150-2000°C. Modifying metals have practically no effect on the nature of the thermal desorption curve for skeletal nickel (50% Al), but change the magnitude of the peaks in different ways, shifting their position mainly to the low-temperature region [4]. The thermal desorption curves clearly show two forms of hydrogen, the desorption of which is carried out in the range of 0-2100°C and 210-6000°C. The first form is desorbed according to the first order equation, and the second form is of the second order. Skeletal FSiCr catalysts exhibit the highest hydrogen capacity, the total volume of desorbed hydrogen up to 6000°C reaches 51.0-55.0 cm³ / g. The sorption capacity of the studied nickel-alumina catalysts occurs through maxima corresponding to 5, 0% content of modifying additives. It should be noted that alloying metals predominantly decrease the concentration of relatively strongly adsorbed forms of H₂, increasing the first weakly adsorbed one. The addition of metals 1.1-1.5 times increases the first form of adsorbed hydrogen.

Table 4.

Thermal desorption of H₂ from skeletal aluminum-nickel catalysts with addition of ferroalloys.

| Catalyst | The volume of desorbed H ₂ from 1 g catalyst (up to 6000°C) cm ³ / g | Selection areas | | | | | |
|-------------|--|---|-----------------------|---------------------|---|-----------------------|---------------------|
| | | I - (0-2100°C) | | | II - (210-6000°C) | | |
| | | Quantity H ₂ , cm ³ / g | T _{max} , °C | Peak edes, kJ / mol | Quantity H ₂ , cm ³ / g | T _{max} , °C | Peak edes, kJ / mol |
| | | H ₂ Me | | | Me \\ H | | |
| Ni (50% Al) | 43.0 | 20.8 | 140 | 34.3 | 21.0 | 250 | 67.9 |
| Ni-3% FSK | 45.5 | 23,7 | 145 | 35.5 | 21.8 | 254 | 68.3 |
| Ni-5% FSK | 44.4 | 24.9 | 130 | 30.8 | 19.5 | 240 | 65.0 |
| Ni-3% FSH | 48.8 | 27.3 | 130 | 30.0 | 21.5 | 235 | 64.0 |
| Ni-5% FSH | 51.5 | 29.6 | 125 | 29.0 | 21.9 | 225 | 63.0 |
| Ni-3% FMo | 53.5 | 33,7 | 115 | 27.6 | 19.8 | 200 | 56.0 |

| | | | | | | | |
|-----------|------|------|-----|------|------|-----|------|
| Ni-5% FMo | 53.0 | 35.8 | 120 | 28.5 | 19.2 | 180 | 53.7 |
|-----------|------|------|-----|------|------|-----|------|

In addition, the first and second forms of adsorbed H₂ are desorbed at relatively low temperatures (I-form - 100-1400C, II-form - 150-2400C) and with a lower Edes value (I-form - 24-30 kJ / mol, II-form - 50.5-65.0 kJ / mol) the amount of weakly adsorbed forms of hydrogen in promoted catalysts reaches 50-60% of the total amount of desorbed H₂. Skeletal Ni-ΦSiCr catalysts exhibit the highest sorption capacity with respect to all forms of H₂. The rest of the catalysts for decreasing the sorption capacity for H₂ are arranged in a row: Ni-FMo> Ni-FSK> Ni-FSK> Ni (50% Al).

So the amount of form of desorbed hydrogen is bound. They are associated with a change in the surface structure of the contact: the ratio of intermetallic nickel (NiAl₁₃ / Ni₂Al₁₃), the presence of surface oxides, which is reflected in the Me - H bond energy.

We have previously determined the structure and adsorption properties of the obtained alloys and catalysts. Subsequently, these catalysts were tested in the process of continuous hydrogenation of benzene in a flow-through column-type unit. During the experiments, the alloys were activated with 10% sodium hydroxide solution. During the first leaching, 30% of aluminum was removed, the catalyst was saturated in a stream of hydrogen for 18 hours at a process temperature of 1600C and a pressure of 0.5 MPa. The benzene feed rate varied from 60 to 120 ml / h. An increase in the hydrogen pressure from 5 to 8 MPa made it possible to reveal that, with an increase in the hydrogen pressure to 6 MPa, the degree of benzene conversion increases, and a further increase in pressure does not affect the catalyst activity.

Table 4 shows that stationary ferroalloy catalysts exhibit absolute activity with respect to cyclohexane and their activity is higher than that of a commercial nickel-titanium catalyst. The values of contact loads overcooked by 30% for aluminum Ni-FMo catalysts at 120°C and 10 MPa reach 0.70 h⁻¹, respectively, which is 2.3 times higher than that of industrial nickel-titanium contacts. The test results showed that the Cu-Al-FMo catalyst is 1.9 times more productive and 2.0 times more stable than the industrial nickel-titanium contact.

Table 5.

Results of continuous hydrogenation of benzene in a flow column unit.

| Catalyst | T°FROM | Catalyst composition | | W h ⁻¹ | Relative continuation Process |
|-----------|---------|----------------------|---------|-------------------|-------------------------------|
| | | Cyclohexane | Benzene | | |
| Ni -Al | 90-100 | 78-85 | 15-22 | 0.12-0.21 | 210 |
| | 110-120 | 81-92 | 8-19 | 0.26-0.28 | |
| | 130-140 | 87-98 | -13 | 0.31-0.35 | |
| Ni-Al-FSK | 110-120 | 94-98 | 2-6 | 0.50-0.64 | 254 |
| | 90-100 | 94-97 | 3-6 | 0.42-0.63 | |
| | 110-120 | 96-99.8 | 0-4 | 0.53-0.70 | |
| Ni-Al-FSH | 110-120 | 98-99.8 | 0-2 | 0.69-0.87 | 410 |
| | 90-100 | 96-99 | 1-4 | 0.47-0.72 | |
| | 130-140 | 99.5-99.8 | 0.5-0.1 | 0.94-1.2 | |
| Ni-Al-FMo | 90-100 | 96-98 | 4-2 | 0.48-0.7 | 420 |
| | 110-120 | 99-99.8 | 1-0.1 | 0.65-0.82 | |
| | 130-140 | 99.5-99.8 | 0.5-0.1 | 0.94-1.2 | |
| Ni-Tiprom | 90-100 | 84-96 | 10-16 | 0.40-0.44 | |

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|--|---------|---------|------|-----------|-----|
| | 40-120 | 84-95 | 5-16 | 0.47-0.50 | 202 |
| | 130-140 | 98-99.8 | 0-10 | 0.50-0.60 | |

CONCLUSION

Thus, we have synthesized for the first time a series of new samples of modified alloyed aluminum-nickel catalysts for the hydrogenation of aromatic hydrocarbons. Their chemical, phase and granulometric compositions, porous structure, adsorption, desorption capacities for hydrogen, as well as the processes of benzene and toluene hydrogenation in their presence have been studied. The promoting effect of ferroalloys (FMo, FSK, FSKh) on the physicochemical and adsorption properties of alloyed aluminum-nickel catalysts, as well as on their catalytic properties in the reaction of liquid-phase hydrogenation has been studied benzene, toluene... The principles of selection of catalysts and modifying additives are proposed. It was found that modifying additives disperse skeletal nickel crystals, increase the specific surface area, the proportion of micropores, the pore volume and their effective radius, as well as the sorption capacity of aluminum-nickel catalysts relatively weakly adsorbed on their surface forms of hydrogen, which contributes to an increase in the catalytic activity of the skeletal catalysts modified by the indicated ferroalloys. A systematic study of the activity of stationary catalysts with addition of ferroalloys in the reaction of continuous catalytic hydrogenation of benzene with a wide variation of the parameters of the technological process has been carried out. At the same time, highly active, stable and selective for cyclohexane new stationary catalysts for industrial purposes operating at temperatures up to 140°C and pressure up to 8 MPa. The Ni-Al-FMo catalyst is recommended for introduction into the production of cyclohexane from benzene [3].

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UDC 664.047:665.3

SIMULATION OF A THREE-QUASI-HARDWARE UNIT FOR OIL-CONTAINING MATERIAL PRESS

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Abstract. *This article analyzes an oil press apparatus, which has found wide application in the production of vegetable oil. Previously, a computer model of the oil extraction process with concentrated parameters was compiled and investigated. Continuing the research, the*