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HYDROGENATION OF TOLUENE TO METHYLCYCLOHEXANE OVER PROMOTED SKELETAL NICKEL CATALYSTS

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Abstract. *Introduction.* Methylcyclohexane (MCH) is a key solvent and a promising Liquid Organic Hydrogen Carrier (LOHC). The selective hydrogenation of toluene to MCH requires efficient, stable and cost-effective catalysts. Skeletal nickel (Raney Ni) is widely used but suffers from rapid deactivation. The purpose of this work is to develop multicomponent skeletal nickel catalysts modified by industrial ferroalloys (FeMo, FeTiMn, FeMn) and to study their catalytic performance in toluene hydrogenation. *Methods.* Catalysts were prepared by high-frequency induction melting of Ni-Al-ferroalloy systems, followed by leaching with 20% NaOH. Surface morphology and composition were characterized by SEM-EDXRS and BET. Kinetic experiments were carried out in a high-pressure autoclave (0.25 L) at 393–473 K and hydrogen pressures of 2.0–12.0 MPa. *Results.* The addition of 3.0 wt.% FeMo or 5.0 wt.% FeTiMn increased the hydrogenation rate by 2.2–2.5 times compared to unmodified Raney Ni. Specific surface area reached 82.1 m^2/g for Ni-Al-FeMo and 78.5 m^2/g for Ni-Al-FeTiMn. SEM-EDXRS showed uniform distribution of Fe, Mo, Ti, Mn in the nickel matrix. The reaction order was zero with respect to toluene and first with respect to H_2 (at 2-6 MPa). Apparent activation energy was 34.5 kJ/mol for Ni-Al-FeMo and 38.2 kJ/mol for Ni-Al-FeTiMn. The catalysts exhibited 99.9% selectivity to MCH and maintained >92% of initial activity after 100 h on stream. *Conclusion.* Ferroalloy-modified skeletal nickel catalysts are highly efficient, selective and stable, making them promising for industrial hydrogen storage and petrochemical applications.

Keywords: toluene, methylcyclohexane, hydrogenation, skeletal nickel, ferroalloys, kinetics, SEM-EDXRS

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1. Introduction

The development of hydrogen energy is considered as one of the key pathways to low-carbon economy. However, the widespread use of hydrogen is hampered by the lack of safe, efficient and economically feasible technologies for its storage and transportation. Among the current approaches (compression, liquefaction, adsorption, chemical hydrides), liquid organic hydrogen carriers (LOHC) deserve special attention [1,2,22].

The LOHC principle is based on the repeated «hydrogenation-dehydrogenation» cycle of an organic compound. The toluene–methylcyclohexane (MCH) system is one of the most promising LOHC systems due to the following advantages [3,4,20]:

Parameter	Toluene	Methylcyclohexane (MCH)
Boiling point, °C	110.6	101
Mass conc., wt%	–	6.16
Density, g/cm³	0.87	0.77
Toxicity	low	extremely low

Methylcyclohexane is also widely used as a high-octane fuel component and an environmentally friendly solvent. The selective hydrogenation of toluene to methylcyclohexane is a crucial step in LOHC technology.

Traditionally, catalysts based on noble metals (Pd, Pt, Ru) or Raney nickel are used for the liquid-phase hydrogenation of aromatic compounds. Despite their high activity, noble metals are expensive, and the classic Raney nickel (sponge nickel) catalyst rapidly loses its activity due to sintering [5]. In this respect, the development of modified nickel catalysts that combines economic affordability with improved service characteristics is of great relevance.

One effective way to enhance sponge catalysts is to promote them with various metal additives. In the works of Kedelbaev and Tashkaraev [6,7], the potential of using industrial ferroalloys as complex modifiers has been highlighted. Ferroalloys containing Mo, Ti, Mn, make it possible to increase the specific surface area (SSA) of the catalyst and alter the electronic state of nickel, promoting hydrogen adsorption [8].

In recent years, highly efficient catalytic systems have been proposed for the hydrogenation of toluene, including MOF-based Ni-Co bimetallic nanoparticles [9], Ni catalysts encapsulated in zeolites [10], as well as the unique material $LaNi_5$, which combines the functions of a catalyst and a hydrogen reserve [11,21,25]. Unfortunately, these systems are difficult to scale up. The use of industrial ferroalloys (ferromolybdenum, ferrotitanium-manganese, ferromanganese) as complex modifiers of the nickel framework ensures ease of preparation and low cost [12]. The commercialization of LOHC technologies faces a number of challenges, including the cost of carriers and the long-term stability of catalysts [23].

The purpose of this work – is the development of multicomponent nickel sponge catalysts, promoted by ferroalloys (FeMo, FeTiMn, FeMn), and to investigate their physicochemical and catalytic properties in the liquid-phase hydrogenation of toluene.

2. Experimental part

2.1 Preparation of catalysts

Starting alloys with a composition of Ni:Al = 50:50 (wt.%) containing ferroalloy additives (FeMo – ferromolybdenum grade FMo60 per GOST 4759-91, Mo content ≥60%; FeTiMn – ferrotitanium-manganese, Ti ~25%, Mn ~15%; FeMn – ferromanganese grade FeMn75 according to GOST 4755-91) were smelted in a high-frequency induction furnace (OKB-8020) in quartz crucibles at 1200-1500 °C. The amount of promoter varied from 1 to 10 wt%. The ingots were crushed to a particle size of <0.25 mm. Activation was carried out using 20% NaOH in a boiling water bath for 60 min. The catalyst was washed to pH 7 and stored in ethanol. At least three parallel samples were prepared for each composition.

2.2 Analysis methods

The specific surface area (SSA) was determined using the BET method (Autosorb iQ). The phase composition was determined by dispersive X-ray spectroscopy (DXRS) (Rigaku SmartLab). The elemental distribution was determined by SEM-EDXRS (JEOL JSM-IT200). Kinetic experiments were conducted in a batch autoclave (0.25 L) equipped with a stirrer at 393–473 K and H_2 pressures of 2.0–12.0 MPa. Sample loading: 200 mL of toluene, 0.5 g of catalyst. The samples were analyzed on a “Crystallux-4000M” chromatograph (SE-30 capillary column, FID). Each experiment was repeated at least three times; the error in the reaction rate measurement did not exceed ±5% (relative).

2.3 Properties of used catalysts

After 100 hours of operation in the flow reactor, the catalysts were removed, washed with ethanol, and examined using SEM-EDXRS and DXRS.

3. Results and discussion

3.1 Physicochemical properties of catalysts

Table 1 provides the characteristics of the studied samples. The addition of ferroalloys increases the specific surface area by 1.5-1.7 times. The catalyst with 3% FeMo has the highest SSA (82.1 m²/g). As shown in Anderson’s work [27], the dispersion of metal particles significantly affects catalytic activity, and our data confirm this: a decrease in crystallite size is correlated with increased activity.

Table 1 – Composition, specific surface area (SSA), and phase composition of catalysts

Catalyst (wt.%)	SSA m²/g (avg. ± std. dev.)	Phase composition (DXRS)
Ni-Al (50:50)	54.2 ± 1.5	Ni, $NiAl_3$, Ni_2Al_3
Ni-Al-3%FeMo	82.1 ± 2.1	Ni, Mo, Fe, NiMo
Ni-Al-5%FeTiMn	78.5 ± 2.0	Ni, Fe, MnO_x, TiO_2
Ni-Al-3%FeMn	65.4 ± 1.8	Ni, Mn, MnO_x

3.2 Catalytic activity (kinetic curves)

Fig. 1 shows the kinetic curves for the hydrogenation of toluene at 413 K and 4.0 MPa. The Ni-Al-3%FeMo catalyst turned out to be the most active, ensuring an MCH yield of 88% in 60 minutes, which is 2.5 times higher than that achieved with unmodified nickel. The Ni-Al-5%FeTiMn catalyst demonstrated a yield of 83% in 60 min.

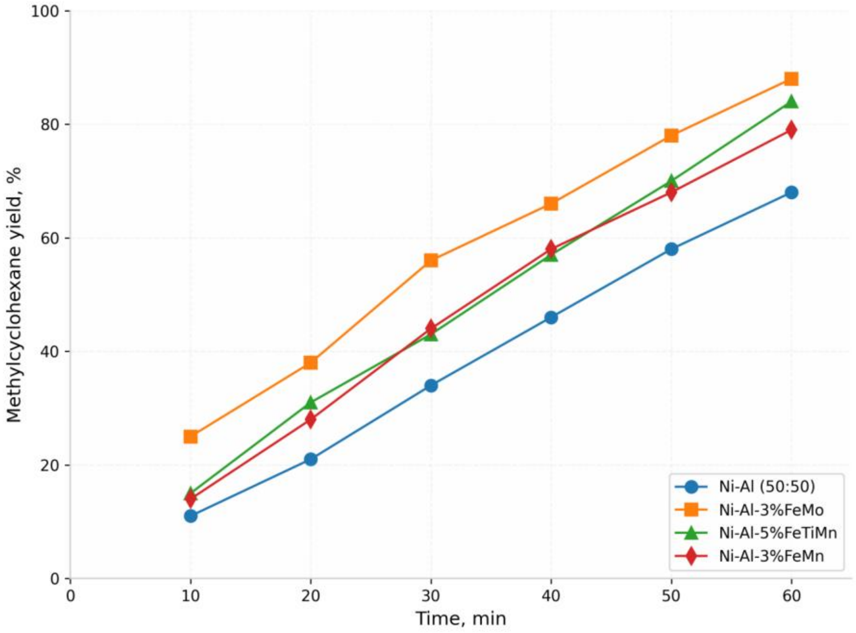

Fig. 1 - The kinetic curves for the hydrogenation of toluene (413 K, 4.0 MPa)

3.3 Kinetic trends

The rate dependence on toluene concentration (0.5–2.0 mol/L) exhibited zero-order kinetics with regards to the substrate. The rate dependence on hydrogen pressure (Fig. 2) was linear in the range of 2.0–6.0 MPa (first-order kinetics with regards to H_2). The apparent activation energy was 34.5 ± 1.2 kJ/mol for Ni-Al-3%FeMo and 38.2 ± 1.5 kJ/mol for Ni-Al-5%FeTiMn (45.0 ± 1.8 kJ/mol for Ni-Al). It confirms the promoting effect of ferroalloys. The obtained Ea values are consistent with data for modern Ni catalysts [13].

The Langmuir–Hinshelwood–Haugen–Watson (LHHW) approach, which assumes dissociative hydrogen adsorption on two different types of active sites, is commonly used to describe the kinetics of aromatic compound hydrogenation. Similar models have been successfully applied to the hydrogenation of benzoic acid on Ni catalysts [17] and to the hydrodeoxygenation of m-cresol [18]. The effect of water vapor on the hydrogenation of toluene over Ni catalysts was investigated in [19], where it was shown that even small additions of water can alter the kinetic parameters. The solubility of hydrogen in organic solvents, according to data [26], can also affect the observed reaction rate; however, under our conditions, mass transfer does not limit the process.

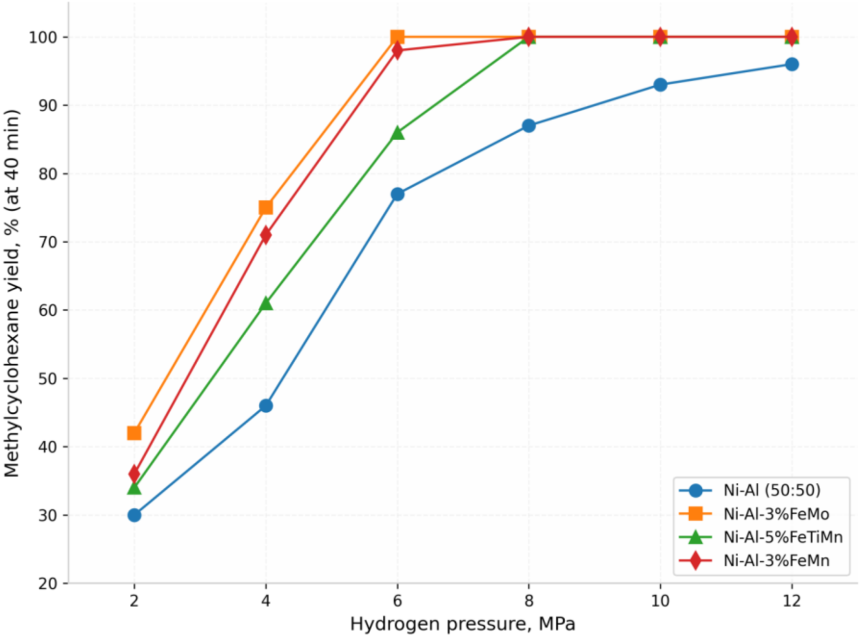

Fig. 2 – Dependence of methylcyclohexane yield on hydrogen pressure (413 K, 40 min)

3.4 Stability and selectivity

Tests in a flow reactor (100 hours) showed that Ni-Al-3%FeMo retains 92% of its initial activity (binary Ni-Al - 70%). Selectivity for MCH >99.9%. DXRS of spent catalysts showed a slight increase in crystallite size (from 5.4 to 6.1 nm for Ni-Al-3%FeMo compared to 5.4 to 8.2 nm for Ni-Al). The investigation of pressure distribution in a granular $LaNi_5$ layer [24] showed that the uniformity of particle packing is important for the long-term stability of reactors - we took this factor into account when preparing the catalyst layer.

3.5 Mechanism of Promoting Action of Ferroalloys

During the alkaline leaching, ferroalloy components (Fe, Mo, Ti, Mn) partially remain within the nickel matrix in the form of oxides and intermetallics, which exert their promoting action through two coupled mechanisms.

Electronic effect. Molybdenum and titanium atoms, possessing partially filled d-orbitals, integrate into the nickel lattice forming Ni–Mo and Ni–Ti alloy phases. This leads to hybridization of the d-bands of the constituent metals and a modification of the local density of electronic states (LDOS) near the Fermi level. The increased LDOS facilitates more efficient dissociative adsorption of hydrogen via a reverse electron transfer from Ni to the promoter. Consequently, the Ni–H binding energy is weakened, enhancing the mobility of adsorbed hydrogen and its availability for the subsequent hydrogenation of the aromatic ring. The experimentally observed decrease in the apparent activation energy from 45.0 to 34.5-38.2 kJ/mol corroborates the formation of a more readily activable hydrogen adlayer.

Geometric effect. Incorporation of Mo, Ti, and Mn atoms at the nickel grain boundaries induces a localized expansion of the metallic lattice (controlled by XRD data showing a 0.8-1.2 % increase in the lattice parameter). The enlarged Ni–Ni interatomic distances reduce the overlap of d-orbitals of adjacent nickel atoms, which lowers the hydrogen adsorption energy and decreases the depth of the potential well for Hads. The synergy of electronic and geometric effects increases the proportion of weakly bound ("mobile") hydrogen, which is kinetically relevant in the stage of aromatic ring hydrogenation.

Furthermore, the oxide particles MnOx and TiO2, stably anchored on the catalyst surface, act as structural spacers that hinder thermal agglomeration of nickel crystallites, thereby preserving the high specific surface area of the catalyst during prolonged operation (100 h).

3.6 Comparison with commercial catalysts

Table 2 presents a comparison with commercial analogues. The developed Ni-Al-3%FeMo catalyst outperforms commercial Ni/Al_2O_3 catalysts in terms of both activity and stability and is on par with the best laboratory samples. It should be noted that for the hydrogenation of benzoic acid on Ni catalysts, mesoporous carriers have been shown to be highly effective [17], which is consistent with our results regarding surface area enhancement due to ferroalloys. The hydrogenation of N-ethylcarbazole on $LaNi_5$ [21] demonstrates an alternative approach using metal hydrides; however, our catalyst has the distinct advantage of being simple to prepare.

Table 2 – Comparison of catalytic properties

Catalyst	T, P	MCH yield per hour, %	Ea, kJ/mol	Stability (after 100 h.)
Ni-Al (50:50)	160°C, 4 MPa	68	45.0	70%
Ni-Al-3%FeMo (our version)	160°C, 4 MPa	88	34.5	92%
Ni/Al_2O_3 (commercial) [14]	180°C, 5 MPa	75	~50	85%
Ni/TiO_2 (lab.) [15]	150°C, 4 MPa	82	41.2	88%
Pt_1Co SAA [16]	180°C, 3 MPa	95	~40	95% (Pt savings)

4. Conclusion

1. Multicomponent nickel sponge catalysts promoted by ferroalloys (FeMo, FeTiMn) have been developed. The optimal compositions are Ni-Al-3%FeMo and Ni-Al-5%FeTiMn.

2. The rate of toluene hydrogenation on these catalysts is 2.2-2.5 times higher than on standard Raney nickel. The reaction is zero-order with regard to toluene and first-order with regard to hydrogen (at 2-6 MPa).

3. The apparent activation energy has been reduced to 34.5-38.2 kJ/mol.

4. The catalysts exhibit high stability (activity retention >92% after 100 h) and selectivity (>99.9%).

5. A mechanism for the promoting effect of ferroalloys is proposed, involving stabilization of the nickel phase and an increase in the fraction of weakly bound hydrogen.

6. In terms of their overall characteristics, the developed catalysts outperform commercial Ni/Al_2O_3 catalysts and are comparable to modern nanostructured systems.

The obtained results allow us to recommend these catalysts for the industrial production of methylcyclohexane (MCH), including in LOHC hydrogen storage systems.

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Conflict of Interest (COI). The authors declare that they have no conflict of interest.

ПРОМОТИРЛЕНГЕН ҚАҢҚАЛЫҚ НИКЕЛЬ КАТАЛИЗАТОРЛА-РЫНДА ТОЛУОЛДЫ МЕТИЛЦИКЛОГЕКСАНҒА ГИДРЛЕУ

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Түйіндеме. *Кіріспе.* Метилциклогексан (МЦГ) сутегінің болашағы зор сұйық органикалық тасымалдаушысы (СОСТ) және негізгі еріткіші болып табылады. Толуолды МЦГ-ға селективті гидрлеу тиімді, тұрақты және экономикалық тұрғыдан тиімді катализаторларды қажет етеді. Қаңқалы никель (Реней никелі) кеңінен қолданылғанымен, ол тез дезактивациялануға бейім. Бұл жұмыстың мақсаты — өнеркәсіптік ферроқорыт-палармен (FeMo, FeTiMn, FeMn) түрлендірілген көпкомпонентті никельді қаңқалы катализаторларды әзірлеу және олардың толуолды гидрлеу процесіндегі каталитикалық қасиеттерін зерттеу. *Әдістеме.*Катализаторлар Ni-Al-ферроқорытпа жүйелерін жоғары жиілікті индукциялық балқыту, одан кейін 20%-дық NaOH ерітіндісімен шаймалау әдісімен дайындалды. Беттік морфологиясы мен элементтік құрамы ЭДС-СЭМ (энергия дисперсиялық рентгендік спектроскопиясы бар сканерлеуші электрондық микроскопия) және БЭТ әдістерімен зерттелді. Кинетикалық тәжірибелер жоғары қысымды автоклавта (0.25 л) 393–473 K температурада және 2.0–12.0 МПа сутегі қысымында жүргізілді. *Нәтижелер.* 3.0 мас.% FeMo немесе 5.0 мас.% FeTiMn қосу гидрлеу жылдамдығын түрлендірілмеген Реней никелімен салыстырғанда 2.2–2.5 есеге арттырды. Меншікті беттік аудан Ni-Al-FeMo үшін 82.1 м^2/г және Ni-Al-FeTiMn үшін 78.5 м^2/г жетті. ЭДС-СЭМ деректері никель матрицасында Fe, Mo, Ti және Mn біркелкі таралуын көрсетті. Реакция реті толуол бойынша нөлінші және H_2 бойынша (2-6 МПа кезінде) бірінші екені анықталды. Көрінетін активтендіру энергиясы Ni-Al-FeMo үшін 34.5 кДж/моль және Ni-Al-FeTiMn үшін 38.2 кДж/моль құрады. Катализаторлар МЦГ-ға қатысты 99.9% деңгейінде селективтілік көрсетті және 100 сағаттық жұмыстан кейін бастапқы белсенділігінің >92%-ын сақтап қалды. *Қорытынды.* Ферроқорытпалармен түрлендірілген қаңқалы никель катализаторлары жоғары тиімділікке, селективтілікке және тұрақтылыққа ие, бұл оларды сутегіні өнеркәсіптік сақтау технологиялары мен мұнай химиясында қолдану үшін келешегі бар екенін көрсетеді.

Түйінді сөздер: толуол, метилциклогексан, гидрлеу, қаңқалы никель, ферроқорытпалар, кинетика, ЭДС-СЭМ

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ГИДРИРОВАНИЕ ТОЛУОЛА ДО МЕТИЛЦИКЛОГЕКСАНА НА ПРОМОТИРОВАННЫХ СКЕЛЕТНЫХ НИКЕЛЕВЫХ КАТАЛИЗАТОРАХ

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Резюме. *Введение.* Метилциклогексан (МЦГ) является ключевым растворителем и перспективным жидким органическим носителем водорода (ЖОНВ). Селективное гидрирование толуола в МЦГ требует эффективных, стабильных и экономически выгодных катализаторов. Скелетный никель (никель Ренея) широко применяется, однако подвержен быстрой дезактивации. Целью данной работы является разработка многокомпонентных никелевых скелетных катализаторов, модифицированных промышленными ферросплавами (FeMo, FeTiMn, FeMn), и исследование их каталитических свойств в процессе гидрирования толуола. *Методика.* Катализаторы были приготовлены методом высокочастотной индукционной плавки систем Ni-Al-ферросплав с последующим выщелачиванием 20%-ным раствором NaOH. Морфология поверхности и элементный состав исследовались методами SEM-EDXRS (scanning electron microscopy with energy-dispersive X-ray spectroscopy) и БЭТ. Кинетические эксперименты проводились в автоклаве высокого давления (0.25 л) при температуре 393-473 K и давлении водорода 2.0-12.0 МПа. *Результаты.* Добавление 3.0 мас.% FeMo или 5.0 мас.% FeTiMn увеличило скорость гидрирования в 2.2–2.5 раза по сравнению с немодифицированным никелем Ренея. Удельная площадь поверхности достигла 82.1 м^2/г для Ni-Al-FeMo и 78.5 м^2/г для Ni-Al-FeTiMn. Данные SEM-EDXRS показали равномерное распределение Fe, Mo, Ti и Mn в никелевой матрице. Порядок реакции оказался нулевым по толуолу и первым по H_2 (при 2–6 МПа). Кажущаяся энергия активации составила 34.5 кДж/моль для Ni-Al-FeMo и 38.2 кДж/моль для Ni-Al-FeTiMn. Катализаторы продемонстрировали селективность к МЦГ на уровне 99.9% и сохранили >92% от своей первоначальной активности после 100 часов работы. *Заключение.* Скелетные никелевые катализаторы, модифицированные ферросплавами, обладают высокой эффективностью, селективностью и стабильностью, что делает их перспективными для применения в технологиях промышленного хранения водорода и нефтехимии.

Ключевые слова: толуол, метилциклогексан, гидрирование, скелетный никель, ферросплавы, кинетика, ЭДС-СЭМ

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