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YTTRIUM AS A MODIFIER IN CATALYSTS FOR SULFUR REMOVAL AND IMPROVED STABILITY

This study delves into the use of yttrium-modified catalysts in hydrotreatment processes, focusing on how they enhance sulfur removal efficiency and improve catalyst stability. By introducing yttrium into various catalyst systems, such as palladium-platinum (Pd-Pt) supported on Y-modified USY zeolite and Y-doped Ni_2P *phosphides, researchers observed significant advancements in catalytic performance. These included better removal of hard-to-treat sulfur compounds like 4,6-dimethyldibenzothiophene (4,6-DMDBT), improved dispersion of active metals, and fewer strong acidic sites, which collectively reduced coke formation.*

The study emphasizes yttrium's unique ability to tackle persistent challenges in hydroprocessing, including the removal of refractory sulfur compounds that conventional catalysts struggle to address. Additionally, yttrium enhanced the structural and operational stability of the catalysts, minimizing coke buildup and maintaining efficiency during extended use. Combining experimental findings with theoretical insights, the research positions yttrium as a pivotal modifier for developing next-generation catalysts capable of achieving ultra-deep desulfurization.

These catalysts not only meet stringent environmental standards but also demonstrate excellent durability, making them ideal for industrial applications, particularly in diesel fuel hydrotreatment. Future work will focus on refining yttrium incorporation methods and scaling these advancements for widespread industrial adoption.

Keywords: Yttrium-modified catalysts, hydrotreatment, ultra-deep desulfurization, palladium-platinum catalysts, USY zeolite, sulfur removal efficiency, coke deposition, hydrogenation.

Introduction

Hydrotreating catalysts are the key of fundamental process not only in diesel hydrotreatment but in all the hydrogenation processes. A catalyst formulation and its preparation play a huge role in their further effectiveness, therefore nowadays plenty variations of structures have been creating, evaluating and implementing. Preparation of catalysts generally follows next steps [1, p. 45]:

– Precipitation;
– Filtration;
– Washing;
– Drying;
– Forming;
– Calcining;
– Imregnation of active metals;
– Activation.

Conventional catalysts like CoMo or NiMo supported on alumina often fail to achieve ultra-deep desulfurization, particularly for refractory sulfur compounds like 4,6-dimethyldibenzothiophene (4,6-DMDBT). These challenges arise due to their steric hindrance and low reactivity under standard hydrotreating conditions. The incorporation of rare earth elements, particularly yttrium, has emerged as an effective strategy to overcome these limitations [2, p. 211].

Yttrium-modified zeolites, such as ion-exchanged Y zeolites, introduce strong Brønsted and Lewis acid sites that enhance the adsorption and removal of nitrogen and sulfur-containing compounds. These catalysts maintain high efficiency even in the presence of competitive adsorbents like toluene, making them particularly valuable for industrial applications [3, p. 5].

The multifunctional nature of yttrium-modified catalysts enables processes like hydrodesulfurization, hydroisomerization, and hydrocracking to occur simultaneously. Polymetallic systems based on ZSM-5 zeolites demonstrate significant improvements in pore structure and active metal dispersion, reducing sulfur content to as low as 0.005 % under optimized conditions [4, p. 814]. Enhanced diffusion properties and improved reactant-product interactions contribute to their superior catalytic performance under mild operating conditions [5, p. 35; 6, p. 12].

Yttrium also stabilizes active phases and modifies the electronic properties of catalyst supports, creating medium-strength acid sites essential for hydrogenation reactions. This stabilization improves the hydrogenation of refractory sulfur compounds like 4,6-DMDBT while sustaining catalyst activity at lower temperatures and pressures, contributing to energy efficiency and process sustainability [7, p. 52; 5, p. 39].

The integration of yttrium into hydrotreating catalysts represents a significant advancement in catalysis, addressing the limitations of conventional systems and aligning with increasingly stringent environmental regulations. By enhancing stability, selectivity, and multifunctionality, yttrium-based catalysts provide a robust pathway for the production of ultra-low sulfur diesel fuels [4, p. 816; 3, 2019, p. 6].

Materials and methods

Production of diesel fuel meeting the requirements of the latest European environmental standards at local refineries will be possible after modernization of existing diesel hydroprocessing units. There are three ways to increase the degree of desulfarization: 1) modernization of the existing hydrotreating units; 2) use of high-efficiency catalysts; 3) introduction of an additional desulfarization unit.

Conversion of sulfur-containing compounds occurs in such processes as oxidative desulfurization, adsorption, extraction, ozonation, ultrasonic action. Modification of catalysts with Yttrium gains plenty positive characteristics which provides it better physical characteristics [8, p. 3032–3043].

The preparation and analysis of the catalysts began with ultra-stable Y-zeolite (USY), chosen for its robust structural and chemical properties. The USY support was modified with yttrium to enhance its performance characteristics. This involved impregnating the zeolite with an aqueous solution of yttrium acetate using the incipient wetness method. After ensuring even distribution of the modifier, the sample was left overnight at room temperature, dried at 110 °C, and then calcined at 300 °C in an oxygen stream. This process stabilized the yttrium within the zeolite structure, with a final yttrium loading of 2.5 wt %.

Following the yttrium modification, palladium and platinum were introduced to the support. Using precursors of $[Pd(NH3)_4]Cl_2$ and $[Pt(NH_3)_4]Cl_2$, the metals were impregnated onto the Y-modified USY to achieve a total metal content of 1.2 wt%, maintaining a Pd-to-Pt molar ratio of 4:1. Once impregnated, the catalyst was dried, calcined, and formed into disks. These were ground into powder and sieved to ensure particle uniformity for reactor testing [9, p. 2].

Characterizing the catalyst was a crucial step to link its physical properties with its performance. Surface area and pore volume were measured using nitrogen adsorption and desorption techniques, while CO chemisorption provided insight into how well the palladium and platinum particles were dispersed across the surface. Acid site distribution and strength were determined by measuring ammonia adsorption heat. Strong acid sites, which can promote unwanted reactions, were minimized due to yttrium's influence, while the overall acid quantity remained stable. Finally, STEM (Scanning Transmission Electron Microscopy) imaging allowed to visually confirm the dispersion of active metals and assess any agglomeration after testing.

The catalytic activity was evaluated through both model and real feedstocks. The model feedstock was a blend of hexadecane and tetralin, with 500 ppm sulfur added as 4,6-dimethyldibenzothiophene (4,6-DMDBT). Real feedstock tests used diesel fuel with 440 ppm sulfur. Reactions were conducted in a fixed-bed flow reactor under high pressure (4.9 MPa) at 280 °C. For model compounds, the space velocity (WHSV) was set at 16 h-1, while for the diesel fuel it was reduced to 4 h-1 to simulate industrial conditions. The reaction outputs were analyzed using gas chromatography equipped with sulfur chemiluminescence detection, allowing precise quantification of sulfur removal and aromatic hydrogenation.

A control catalyst using unmodified USY was prepared and tested under the same conditions to highlight the effects of yttrium modification. This comparison was essential in demonstrating how yttrium enhanced both the stability and efficiency of the Pd-Pt catalyst, particularly in addressing challenging sulfur compounds like 4,6-DMDBT [10, p. 2].

The catalytic performance was tested using both model and real diesel feedstocks. The model feedstock consisted of tetralin and hexadecane mixed with 500 ppm 4,6-dimethyldibenzothiophene (4,6-DMDBT) as the sulfur-containing compound. The tests were carried out in a fixed-bed reactor at 280 °C under 4.9 MPa hydrogen pressure, with a weight hourly space velocity (WHSV) of 16 h-1, and a hydrogen-to-oil ratio of 500 NL/L. For real feedstock testing, desulfurized diesel fuel with 440 ppm sulfur was used. These conditions simulated typical industrial hydrodesulfurization operations.

The outlet gases from the reactor were analyzed for sulfur content and aromatic composition using gas chromatography coupled with sulfur chemiluminescence detection (GC-SCD). This allowed for precise evaluation of sulfur removal efficiency and aromatic hydrogenation activity.

Table 1 – Properties of Pd-Pt/Y-USY and Pd-Pt/USY [8]

Catalyst	Surface area		Dispersion	Acidic properties	
	Total [m^2/g]	Mesopore [m^2/g]		Initial heat of adsorption [kJ/mol]	Acid amount [mmol/g]
Pd-Pt/Y-USY	465	58	0.56	119	0.18
Pd-Pt/USY	484	52	0.50	126	0.19

Table 1 presents the properties of the two catalysts, Pd-Pt/Y-USY and Pd-Pt/USY. It shows that yttrium modification of the USY zeolite slightly reduced the total surface area of the catalyst from 484 m^2/g for Pd-Pt/USY to 465 m^2/g for Pd-Pt/Y-USY. However, the mesopore surface area increased from 52 m^2/g to 58 m^2/g showing that yttrium modification improves the pore structure, potentially enhancing the catalyst's accessibility to reactants.

The effect of yttrium modification was compared to a control catalyst, prepared using unmodified USY, under identical conditions. The comparative tests allowed to assess how yttrium-modification improved catalyst stability, sulfur resistance, and overall catalytic performance.

Results and discussion

Summarizing the experiments with Yttrium modified catalyst several characteristics were analyzed and represented. The first aim is to define the amount of sulfur as a main contaminant in catalysts comparing with a feedstock and characterize an impact of implementation of Yttrium modifiers into the catalyst.

Table 2 compares the properties of the feedstock and the hydrotreated products obtained using both the Pd-Pt/USY and Pd-Pt/Y-USY catalysts. The feedstock used for testing contained 440 ppm sulfur and 26.8 wt% total aromatics, with 5.4 wt% consisting of multi-ring aromatic compounds. After treatment with the Pd-Pt/Y-USY catalyst, the sulfur content in the hydrotreated product dropped to 16.8 ppm after 48 hours, representing a sulfur removal efficiency of 96.2 %. After 216 hours of operation, the sulfur content was reduced to 28.5 ppm, and the total aromatic content increased slightly to 8.3 wt%. However, despite this slight increase, the Pd-Pt/Y-USY catalyst maintained a high sulfur removal rate of 93.5 %, demonstrating its long-term stability and effectiveness.

Table 2 – Properties of Feedstock and Hydrotreated Product

Properties	Feedstock	Hydrotreated product			
		Pd-Pt/Y-USY		Pd-Pt/USY	
		48 h	216 h	48 h	216 h
Boiling point [°C]					
IBP	211	180	185	159	183
10%	244	231	236	210	233
50%	286	276	280	272	279
90%	354	349	351	348	351
EP	387	382	384	381	384
Sulfur [wtppm]	440	16.8	28.5	17.4	47.9
Total aromatics [wt%]	26.8	4.9	8.3	7.6	11.9
Mono-ring aromatics [wt%]	21.4	4.3	7.1	7.0	10.5

2+-ring aromatics [wt%]	5.4	0.6	1.2	0.6	1.4
HDS [%]	-	96.2	93.5	96.0	89.1
HAD [%]	-	81.6	68.9	71.7	55.7

To be convinced and study collected data deeply GC-SCD chromatography was carried out. The figure will ensure to compare data and detailly demonstrate a rate of removing contaminants after hydrotreatment.

Figure 1 in the study presents the gas chromatography with sulfur chemiluminescence detection (GC-SCD) chromatograms for both the feedstock and the hydrotreated products after 216 hours of operation. The chromatograms provide a detailed analysis of sulfur-containing compounds in the diesel fuel before and after treatment with the Pd-Pt/Y-USY catalyst.

In the feedstock chromatogram, the presence of sulfur compounds, particularly 4,6-dimethyldibenzothiophene (4,6-DMDBT), is clearly visible. These sulfur compounds are challenging to remove and are typically considered refractory sulfur species in fuel. The peaks representing sulfur compounds are prominent, confirming that the sulfur content of the diesel fuel is relatively high, around 440 ppm.

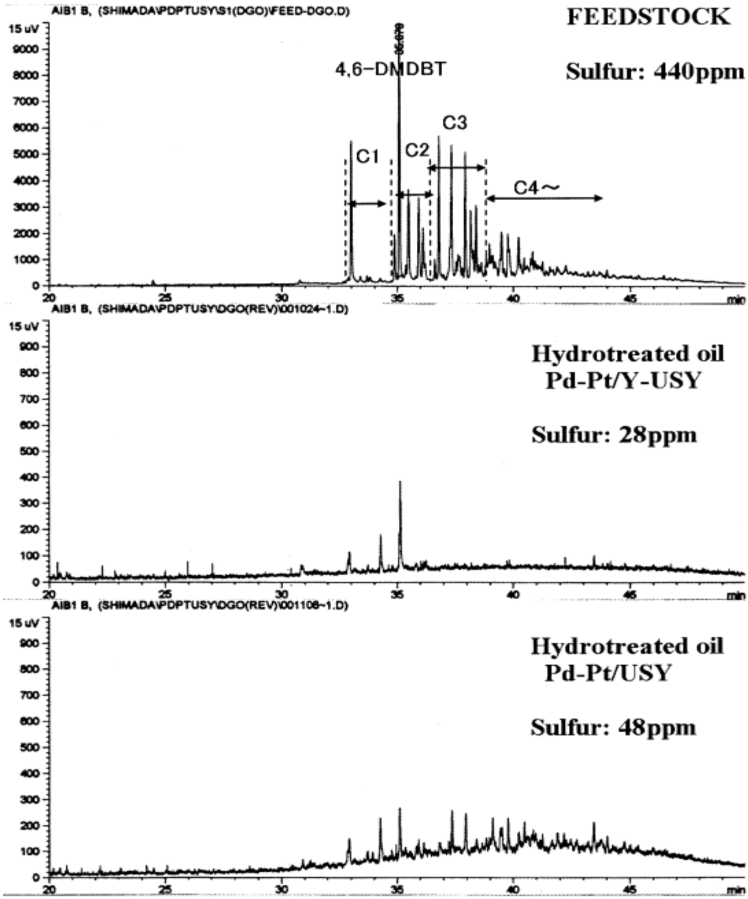

Figure 1 – GC-SCD Chromatograms of Feedstock
and Hydrotreated Product [8]

In contrast, the hydrotreated product chromatogram shows a significant reduction in sulfur peaks after 216 hours of operation, particularly for the 4,6-DMDBT and other refractory sulfur compounds. This dramatic decrease in the sulfur peaks in the chromatogram directly correlates with the sulfur removal data presented in Table 2, where the Pd-Pt/Y-USY catalyst reduced the sulfur content of the treated fuel to 28.5 ppm after the prolonged treatment period. The reduction in sulfur compounds seen in the chromatogram clearly demonstrates the high effectiveness of the Pd-Pt/Y-USY catalyst in removing sulfur from the fuel, particularly from the more stubborn, difficult-to-remove sulfur species like 4,6-DMDBT.

The incorporation of yttrium into catalysts significantly enhanced their catalytic performance, including activity and stability. For example, Pd-Pt/Y-USY (Yttrium-modified Ultra-stable Y zeolite) achieved 96.2 % sulfur removal from model feedstocks and maintained over 93 % efficiency after 216 hours. Additionally, Yttrium-modified Ni2P demonstrated a 5.4 % higher hydrodesulfurization (HDS) conversion rate compared to its unmodified counterpart. The introduction of yttrium also reduced the number of strong acid sites, thereby mitigating excessive hydrocracking and coke deposition. Notably, Pd-Pt/Y-USY exhibited only 2.73 wt% coke deposition compared to 3.65 wt% for unmodified catalysts, highlighting the stabilizing effect of yttrium. A table below shows it in details to provide a clearer comparison and description [9, 10].

Table 3 – Properties of Pd-Pt/Y-USY vs. Pd-Pt/USY Catalysts

Catalyst	Surface Area (m^2/g)	Acid Amount (mmol/g)	Coke Deposit (wt%)
Pd-Pt/Y-USY	465	0.18	2.73
Pd-Pt/USY	484	0.19	3.65

The effectiveness of yttrium-modified catalysts is illustrated in two complementary figures, providing a comprehensive understanding of their performance advantages. Figure 2 compares the performance of Pd-Pt/Y-USY and Pd-Pt/USY catalysts in removing sulfur from 4,6-DMDBT over a continuous operation of 216 hours. Initially, both catalysts exhibit high sulfur removal efficiencies, but as time progresses, a clear divergence emerges. Pd-Pt/Y-USY consistently maintains its efficiency above 93 %, demonstrating its superior stability and resistance to deactivation. In contrast, the unmodified Pd-Pt/USY catalyst shows a steady decline in sulfur removal, highlighting the limitations of conventional catalysts under prolonged operation.

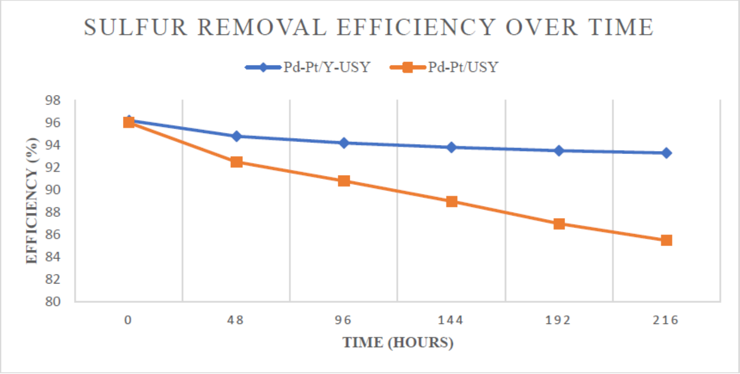

Figure 2 – Sulfur removal efficiency over time

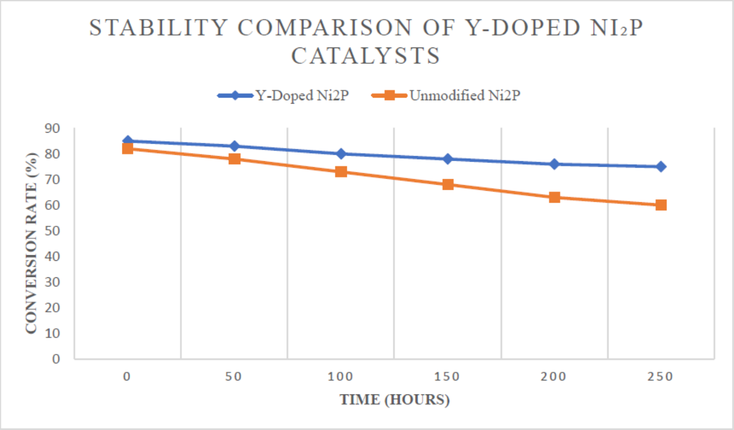

Figure 3 – Stability comparison of Y-doped Ni2P catalysts

This notable difference in stability can be attributed to the introduction of yttrium, which reduces strong acid sites responsible for coke formation and ensures better dispersion of active metals across the support surface. The sustained performance of Pd-Pt/Y-USY underscores its reliability for ultra-deep desulfurization processes, particularly in treating refractory sulfur compounds like 4,6-DMDBT.

Building on these findings, the figure 3 emphasizes the role of yttrium in enhancing catalyst performance. The graph illustrates the conversion rates of

Y-doped Ni2P and unmodified Ni2P catalysts during hydrodesulfurization (HDS) of dibenzothiophene over an extended reaction period. The Y-doped catalyst exhibits a consistently higher conversion rate, approximately 5.4 % greater than its unmodified counterpart. This performance enhancement can be linked to yttrium's ability to stabilize the Ni2P active phase, preventing phase transformation and structural degradation during prolonged operation.

Yttrium-modified Ni2P catalysts demonstrated stable hydrodesulfurization (HDS) performance during prolonged operational periods. This enhanced stability is attributed to their remarkable resistance to coke formation, as confirmed by detailed morphological analyses conducted using scanning electron microscopy (SEM). Moreover, the incorporation of yttrium significantly improved the structural properties of the catalysts, promoting better dispersion of active phases. This, in turn, ensured the uniform distribution and accessibility of active sites, thereby contributing to the sustained catalytic efficiency.

Conclusion

The results of this study demonstrate that yttrium-modified catalysts offer significant advantages in hydrotreatment processes. By enhancing the structural and chemical properties of traditional catalysts, yttrium incorporation leads to improved sulfur removal efficiency, particularly for refractory compounds such as 4,6-DMDBT. Moreover, the stability of these catalysts, even under extended operating conditions, underscores their potential to overcome challenges like coke deposition and deactivation. The consistent performance of Pd-Pt/Y-USY and Y-doped Ni_2P catalysts highlights their suitability for industrial applications where ultra-deep desulfurization is required. Future research should focus on scaling up these findings to industrial levels, optimizing yttrium incorporation methods, and evaluating the long-term economic and environmental impacts of these modifications [8].

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КҮКІРТТІ ЖОЮ ЖӘНЕ ТҰРАҚТЫЛЫҚТЫ АРТТЫРУ ҮШІН КАТАЛИЗАТОРЛАРДАҒЫ МОДИФИКАТОР РЕТІНДЕГІ ИТТРИЙ

Бұл зерттеу гидротазарту процестерінде иттриймен модификацияланған катализаторларды қолдануға арналды, олардың күкіртті жою тиімділігін арттыруға және катализаторлардың тұрақтылығын жақсартуға әсерін зерттеуге баса назар аударды.

Иттрийді әртүрлі каталитикалық жүйелерге, атап айтқанда, иттриймен модификацияланған USY цеолитіндегі палладий-платина (Pd-Pt) және иттрий қосылған Ni_2P *фосфидтеріне енгізу арқылы каталитикалық тиімділіктің едәуір жақсарғаны байқалды. Мұндай жетістіктерге 4,6-диметилдибензотиофен (4,6-DMDBT) сияқты күрделі күкірт қосылыстарын тиімді жою, белсенді металдардың дисперсиясын арттыру және күшті қышқыл орталықтарының санын азайту арқылы кокстың түзілуін төмендету жатады.*

Зерттеу иттрийдің дәстүрлі катализаторлар шеше алмайтын тұрақты күкірт қосылыстарын жою сияқты гидротазарту процестеріндегі негізгі мәселелерді шешудегі ерекше қабілетін көрсетеді. Сонымен қатар, иттрий катализаторлардың құрылымдық және пайдалану тұрақтылығын жақсартып, кокстың пайда болуын азайтады және ұзақ уақыт бойы тиімділігін сақтайды. Эксперименттік деректер мен теориялық тұжырымдарды біріктіре отырып, бұл зерттеу иттрийді ультратерең десульфуризацияға арналған жаңа буын катализаторларын әзірлеудегі негізгі модификатор ретінде таниды.

Бұл катализаторлар қатаң экологиялық талаптарға ғана сәйкес келіп қоймай, жоғары тұрақтылығын да көрсетеді, сондықтан оларды өнеркәсіптік қолдану, әсіресе дизель отынын гидротазарту үшін өте қолайлы етеді. Болашақта жұмыс иттрийді енгізу әдістерін оңтайландыруға және осы нәтижелерді кең ауқымды өнеркәсіптік қолдану үшін масштабтауға бағытталатын болады.

Кілтті сөздер: иттриймен модификацияланған катализаторлар, гидротазарту, ультратерең десульфуризация, палладий-платина катализаторлары.

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ИТТРИЙ КАК МОДИФИКАТОР В КАТАЛИЗАТОРАХ ДЛЯ УДАЛЕНИЯ СЕРЫ И ПОВЫШЕНИЯ СТАБИЛЬНОСТИ

Это исследование посвящено использованию катализаторов, модифицированных иттрием, в процессах гидроочистки, с акцентом на повышение эффективности удаления серы и улучшение стабильности катализаторов. Введение иттрия в различные каталитические системы, такие как палладий-платина (Pd-Pt), нанесённые на USY цеолит, модифицированный иттрием, и фосфиды Ni_2P, легированные иттрием, позволило добиться значительных улучшений каталитической активности. Среди достижений можно выделить более эффективное удаление трудноудаляемых серосодержащих соединений, таких как 4,6-диметилдибензотиофен (4,6-DMDBT), улучшение дисперсии активных металлов и уменьшение количества сильных кислотных центров, что в совокупности привело к снижению образования кокса.

Исследование подчёркивает уникальную способность иттрия решать ключевые проблемы гидроочистки, включая удаление устойчивых серосодержащих соединений, с которыми не справляются традиционные катализаторы. Более того, иттрий усиливает структурную и эксплуатационную стабильность катализаторов, снижая образование кокса и поддерживая эффективность в течение длительного времени. Комбинируя экспериментальные данные и теоретические выводы, исследование демонстрирует потенциал иттрия как ключевого модификатора для разработки катализаторов нового поколения, способных обеспечить ультраглубокую десульфуризацию.

Эти катализаторы не только соответствуют строгим экологическим стандартам, но и демонстрируют высокую долговечность, что делает их идеальными для промышленных применений, в частности, для гидроочистки дизельного топлива. В будущем работа будет сосредоточена на оптимизации методов введения иттрия и масштабировании полученных результатов для более широкого промышленного использования.

Ключевые слова: катализаторы, модифицированные иттрием, гидроочистка, сверхглубокая десульфурация, палладий-платиновые катализаторы, USY цеолит, эффективность удаления серы, образование кокса, гидрирование.

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