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STUDY OF CATALYSTS BASED ON MESOPOROUS ALUMINOSILICATE IN THE DEAROMATIZATION PROCESS OF A MODEL MIXTURE: PHYSICO-CHEMICAL CHARACTERISTICS AND CATALYTIC ACTIVITY

Annotation: The limitation on the content of aromatic hydrocarbons and sulfur is associated with environmental requirements according to Euro-5 standards. Upon combustion, these substances release highly toxic compounds into the atmosphere, adversely affecting human health and the surrounding environment. Additionally, a high concentration of aromatic hydrocarbons in diesel fuel significantly degrades its quality, leading to soot formation and reducing its cetane number. According to Euro-5 standards, the content of polycyclic aromatic hydrocarbons and sulfur compounds should not exceed 8% and 10 mg/kg, respectively. To address the challenge of producing high-quality diesel fuels, the hydrotreatment process is employed in the presence of heterogeneous catalysts.

The article presents the synthesis of mesoporous aluminosilicates and bifunctional catalysts based on them to study the activity of the hydrotreatment process for a model mixture. The obtained samples were investigated using various physico-chemical methods such as SEM, low-temperature nitrogen adsorption/desorption, XRD, and FT-IR. It was established that the Ni/MAS-H-bentonite and Mo/MAS-H-bentonite catalysts exhibit high activity and selectivity in the hydrotreatment process under optimal conditions (240°C, 3 MPa, and 0.5 h⁻¹).

Key words: mesoporous aluminosilicates, bifunctional catalysts, nickel-molybdenum-containing catalysts, hydrodearomatization, model mixture.

Introduction

Global energy consumption is increasing, and requirements for diesel fuel are becoming more stringent worldwide year by year. These requirements include limitations not only on the content of aromatic hydrocarbons and sulfur but also on the cetane number [1]. According to Euro-5 standards, the content of polycyclic aromatic hydrocarbons and sulfur compounds should not exceed 8% and 10 mg/kg, respectively [2]. Upon combustion, these substances release highly toxic compounds into the atmosphere, such as carbon monoxide, sulfur dioxide, and soot [3].

To address the challenge of producing high-quality diesel fuels, the process of hydrodearomatization is employed in the presence of heterogeneous catalysts. During the process, aromatic compounds transform into cycloalkanes, thereby increasing the cetane number and enhancing the low-temperature properties of diesel fuel [4]. Moreover, dearomatization is not only used to obtain high-quality diesel fuel but also for the synthesis of reactive fuels, leading to an increase in the volume of produced fuel, and various environmental issues arise due to the impacts on the environment [5].

The main mechanism of this process is the transformation of large molecules into smaller, more active catalyst centers [6]. Traditional porous carriers for the hydrodearomatization process include zeolites, mesoporous aluminosilicates, and γ -Al₂O₃. However, the strong acidic centers of zeolites and the low specific surface area, weak acidic centers, and unstructured pores of γ -Al₂O₃ are their main disadvantages. Therefore, structured mesoporous aluminosilicates are considered

promising carriers for catalysts because of the following advantages: a) distinct structure; b) high specific surface area; c) mesopore sizes [7].

Currently, two-stage hydrodearomatization processes have been developed for the production of Euro-5 standard diesel fuel. In the first stage, at temperatures of 340–380°C and pressures of 50–70 atm, heteroatomic compounds are removed on sulfide catalysts Co-Mo and Ni-Mo containing γ - Al_2O_3 /zeolite. Simultaneously, the partial hydrogenation reaction of di- and polyaromatic hydrocarbons to monoaromatics takes place. As a result, the content of aromatic hydrocarbons and sulfur is reduced to 25 wt.% and 30–100 ppm, respectively. In the second stage, the feed undergoes further hydrodearomatization at 260–300°C and pressures up to 70 atm on catalysts γ - Al_2O_3 promoted with metals Pt and/or Pd. It should be noted that for the Pt-Pd/Al-SBA-15(5)/ γ - Al_2O_3 catalyst, the optimal temperature for hydrodearomatization of 2-methylnaphthalene in n-heptane is 260°C with a selectivity of 97% to methyltetralin [8].

The results of hydrodearomatization experiments on Ni-Mo and Ni-W catalysts indicate that at low values of the feedstock volumetric flow rate (less than 2 h⁻¹) and high hydrogen pressure, a low content of aromatic compounds is achieved in the hydrodearomatization products. The authors concluded that at high volumetric flow rates and temperatures below 350°C, the concentration of monoaromatic hydrocarbons in the final product would be significantly higher than in the feedstock. This is attributed to the fact that diaromatic hydrocarbons will undergo hydrogenation to form monoaromatic compounds [9].

The purpose of this article is to investigate the activity of bifunctional catalysts based on mesoporous aluminosilicate in the process of hydrodearomatization of a model mixture.

Materials and Methods

Mesoporous aluminosilicates were synthesized using the templating method with hexadecylamine as the structure-directing agent. The synthesis of bifunctional catalysts was carried out by the wet impregnation method, using pre-activated bentonite from the Tagan deposit in the East Kazakhstan region as the substrate for the catalysts. Nickel nitrate and ammonium heptamolybdate tetrahydrate were chosen as precursors for Ni and Mo, respectively. Each sample of the catalyst was prepared and measured in triplicate.

The morphology of the synthesized mesoporous aluminosilicate was determined using scanning electron microscopy with Hitachi TM-1000.

The porous structure of the synthesized samples and BET surface area values were investigated using the standard nitrogen adsorption/desorption method at 77 K on Micromeritics TriStar 3000 equipment (Micromeritics, Norcross, Georgia, USA).

The mesoporous structure of the synthesized samples was studied by Fourier-transform infrared spectroscopy using the Nicolas iS50 FT-IR and wide-angle X-ray scattering on the Philips X'PERT MPD diffractometer (Philips, Eindhoven, Netherlands).

The catalytic properties of bifunctional catalysts were tested in a fixed-bed flow reactor in a stream of hydrogen under the following process conditions: 220–300°C, 3 MPa, feedstock volumetric flow rate of 0.5 h⁻¹. A solution of 2-methylnaphthalene in n-hexadecane was used as a model hydrocarbon since 2-methylnaphthalene is a typical representative of diaromatic hydrocarbons in the diesel fraction.

Results and Discussion

Figures 1 and 2 illustrate schemes for the synthesis of mesoporous aluminosilicate and bifunctional catalysts based on it. In our previous work [10], the authors presented a comprehensive description of the synthesis methodology for mesoporous aluminosilicate and the bifunctional catalyst based on it.

The SEM image (Figure 3) reveals numerous homogeneous particles of a round shape with an average diameter of 610 nm and ordered hexagonal arrays of mesopores with uniform pore sizes. Such a diameter is characteristic of mesoporous material, indicating the mesoporosity of the obtained sample [11–13].

The synthesized samples' low-temperature N₂ adsorption/desorption isotherms are of type IV with a more pronounced hysteresis loop closer to H4 according to the IUPAC classification (Figure 4a). Type IV isotherms demonstrate a narrow pore size distribution in the mesopore range [14]. Additionally, the hysteresis loop at relative pressures exceeding $P/P_0=0.4$ is characteristic of mesoporous materials. Capillary condensation in mesopores explains the presence of a hysteresis loop at low pressures.

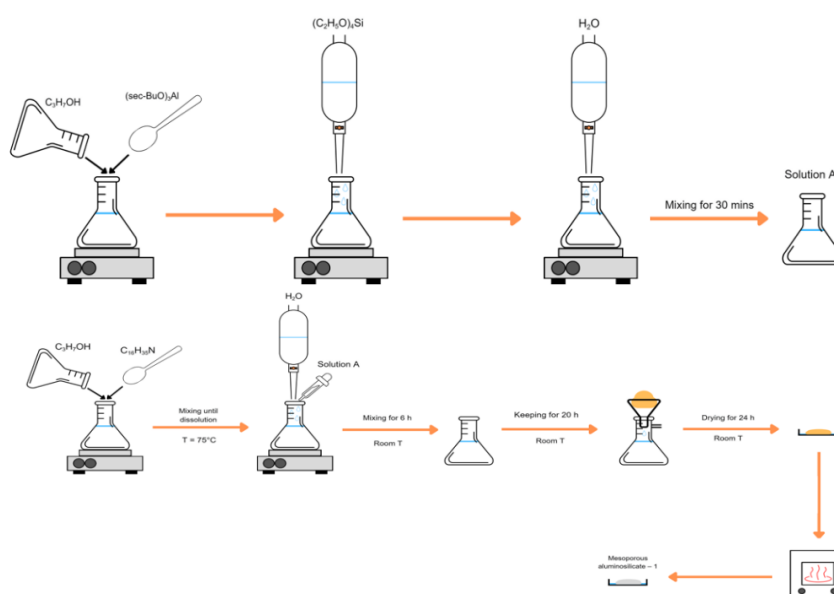


Figure 1 – Synthesis of mesoporous aluminosilicate (MAS)

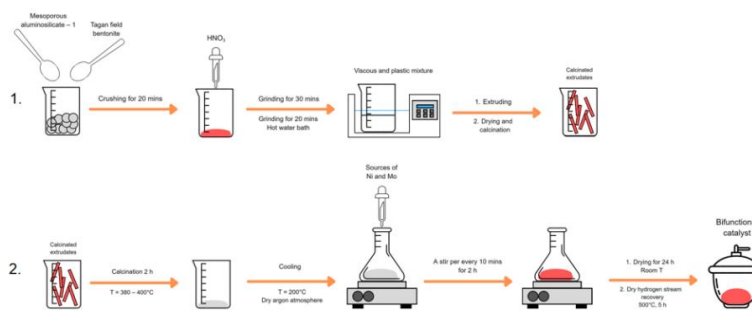


Figure 2 – Synthesis of bifunctional catalysts based on MAS

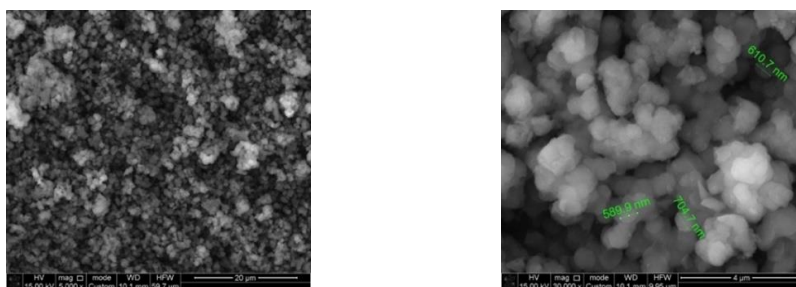


Figure 3 – SEM images of mesoporous aluminosilicate

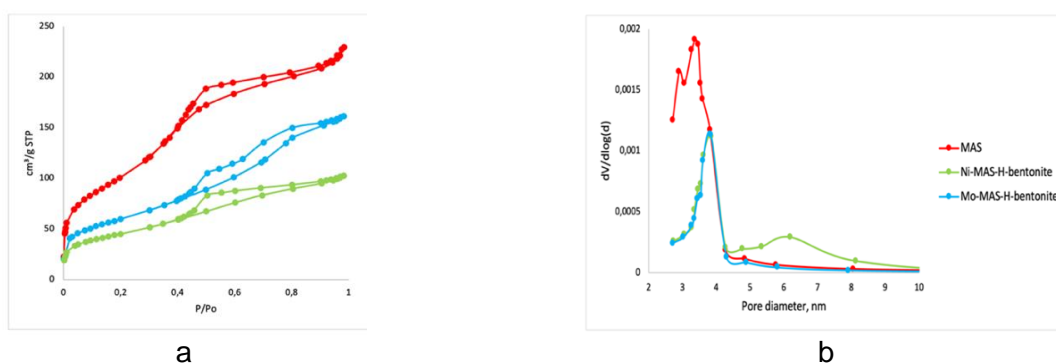


Figure 4 – Low-temperature nitrogen adsorption/desorption isotherms (a) and pore size distribution (b) of mesoporous aluminosilicate (MAS) and bifunctional catalysts based on it. Error bars depict 95% confidence intervals

The pore size distribution is presented in Figure 4b and was determined using the BJH (Barrett-Joyner-Halenda) method. It was found that all synthesized samples exhibit bimodal characteristics in the pore size distribution [15]. The results of specific surface area, pore volume, and average mesopore diameter of the synthesized materials are presented in Table 1. There was a decrease in all the mentioned physico-chemical characteristics, likely associated with the partial blocking of the aluminosilicate pore system by Ni and Mo [16].

Table 1 – Physico-chemical properties of mesoporous aluminosilicate (MAS) and bifunctional catalyst Ni/MAS-H-bentonite and Mo/MAS-H-bentonite

Samples	Specific surface area, m ² /g	Pore volume, cm ³ /g	Average diameter pore, nm
MAS	385.5	0.3721	17.01
Ni/MAS-H-bentonite	281.2	0.2833	15.62
Mo/MAS-H-bentonite	298.7	0.2392	16.03

The wide-angle X-ray diffraction method confirms the amorphous structure of mesoporous aluminosilicate (Figure 5a). The broad halo in the 2 θ range from 40° to 60° is characteristic of the amorphous nature of the sample, and the absence of distinct peaks indicates that the molecules of mesoporous aluminosilicate do not crystallize [16-19]. X-ray peaks of bifunctional catalysts (Ni/MAS-H-bentonite and Mo/MAS-H-bentonite) reveal a crystalline structure without any traces of amorphous material (Figure 5b).

Additionally, the structure of synthesized mesoporous aluminosilicates was studied using FT-IR spectroscopy (Figure 6). The corresponding peaks observed at 1059 cm⁻¹ indicate the presence of asymmetric stretching vibrations of Si-O-Si and Al-O-Al. Peaks in the range of 789-807 cm⁻¹ suggest the presence of crystalline ordering, attributed to the internal vibration of tetrahedra containing Al and Si. The IR spectra below 500 cm⁻¹ is associated with Si-O-Si oscillations. A peak at 1630 cm⁻¹, associated with the O-H bond, is explained by the presence of Al and Si hydroxide particles, along with H₂O [17, 21-23].

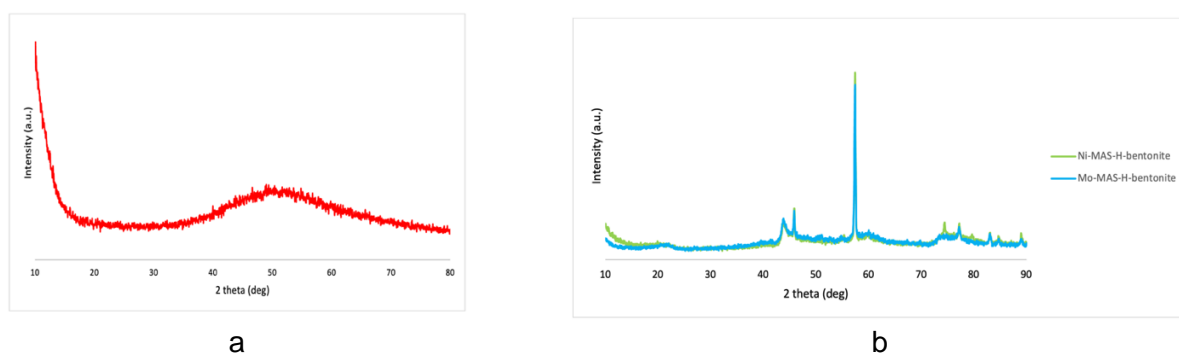


Figure 5 – X-ray diffraction of wide-angle scattering of mesoporous aluminosilicate (a) and bifunctional catalysts based on it. Error bars depict 95% confidence intervals

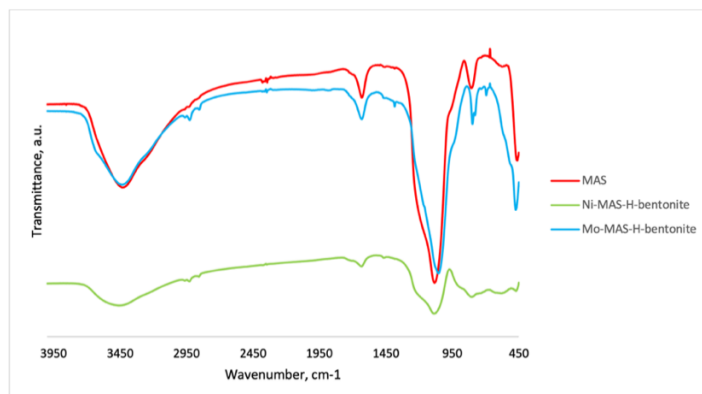


Figure 6 – FT-IR spectra of mesoporous aluminosilicate (MAS) and bifunctional catalysts based on it. Error bars depict 95% confidence intervals

The catalytic activity of the synthesized catalysts in the hydrotreatment process is provided in Table 2. Based on the obtained experimental data, it is evident that the selectivity towards 6-methyltetralin is higher than towards 2-methyltetralin for all samples. This can be attributed to the fact that the aromatic ring with a methyl substituent is less readily hydrogenated than the unsubstituted ring due to steric effects [24, 25].

The dependence of product selectivity in the hydrotreatment process and conversion of 2-methylnaphthalene on a Ni/MAS-H-bentonite catalyst as a function of temperature is presented in Figure 7.

Table 2 – Influence of temperature on the hydrogenation process on Ni/MAS-H-bentonite and Mo/MAS-H-bentonite catalysts for the model compound n-hexadecane+2-methylnaphthalene (3 MPa, feedstock volumetric flow rate of 0.5 h⁻¹)

Parameters	Ni/MAS-H-bentonite					Mo/MAS-H-bentonite				
	Temperature, °C									
	220	240	260	280	300	220	240	260	280	300
Conversion, %	91	91	95	95	95	89	90	91	92	92
Selectivity, %										
2-MD	35	90	70	23	49	29	76	55	12	32
2-MT	23	5	10	32	19	10	2	14	47	47
6-MT	42	5	20	45	32	61	22	31	41	21

*2-MD – 2 methyldecaline, 2 MT – 2-methyltetraline, 6 MT – 6 methyltetralyne.

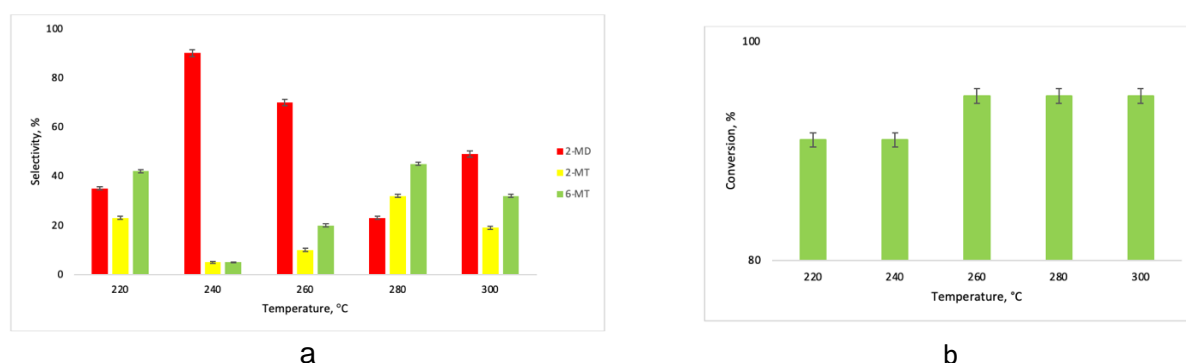


Figure 7 – Dependence of the selectivity of products in the hydrotreating process (a) and the conversion (b) of 2-methylnaphthalene on Ni/MAS-H-bentonite catalyst on temperature. Error bars depict 95% confidence intervals

The selectivity towards 2-methyldecalines at 240 °C is 90%, then it decreases with an increase in temperature up to 280 °C, after which it slightly increases again. The optimal temperature for hydrogenation on the specified catalyst can be considered as 240°C, where the conversion is close to quantitative, and the content of fully hydrogenated products (2-methyldecalins) reaches a maximum value of 91%.

Conclusion

Thus, an ordered mesoporous aluminosilicate was synthesized using the template method. Physico-chemical analytical techniques were applied to the obtained samples. By using scanning electron microscopy, low-temperature nitrogen adsorption/desorption, wide-angle X-ray diffraction, FT-IR spectroscopy, it has been verified that aluminosilicates and catalysts based on them have an ordered and mesoporous structure. The catalytic activity of Ni/MAS-H-bentonite and Mo/MAS-H-bentonite was studied in the hydrotreatment process of a model compound. It was demonstrated that bifunctional catalysts exhibit sufficient activity and selectivity in the hydrotreatment process of n-hexadecane+2-methylnaphthalene under optimal conditions of 240°C, 3 MPa, and 0.5 h⁻¹.

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ИССЛЕДОВАНИЕ КАТАЛИЗАТОРОВ НА ОСНОВЕ МЕЗОПОРИСТОГО АЛЮМОСИЛИКАТА В ПРОЦЕССЕ ДЕАРОМАТИЗАЦИИ МОДЕЛЬНОЙ СМЕСИ: ФИЗИКО-ХИМИЧЕСКИЕ ХАРАКТЕРИСТИКИ И КАТАЛИТИЧЕСКАЯ АКТИВНОСТЬ

Ограничение на содержание ароматических углеводородов и серы связано с требованиями экологических стандартов Евро-5. При сгорании эти вещества высвобождают высокотоксичные соединения в атмосферу, негативно воздействуя на здоровье человека и окружающую среду. Кроме того, высокая концентрация ароматических углеводородов в дизельном топливе существенно снижает его качество, приводя к образованию сажи и уменьшению цетанового числа. Согласно стандартам Евро-5, содержание полициклических ароматических углеводородов и соединений серы не должно превышать 8% и 10 мг/кг соответственно. Для решения задачи производства высококачественного дизельного топлива используется процесс гидроочистки в присутствии гетерогенных катализаторов.

В статье представлен синтез мезопористых алюмосиликатов и бифункциональных катализаторов на их основе для изучения активности процесса гидроочистки на модельной смеси. Полученные образцы исследовались с использованием различных физико-химических методов, таких как СЭМ, адсорбция/десорбция азота при низкой температуре, рентгеноструктурный анализ и Фурье-преобразование инфракрасного спектра. Было установлено, что катализаторы Ni/MAS-H-бентонит и Mo/MAS-H-бентонит проявляют высокую активность и селективность в процессе гидроочистки при оптимальных условиях (240°C, 3 МПа и 0,5 ч⁻¹).

Ключевые слова: мезопористые алюмосиликаты, бифункциональные катализаторы, никель-молибденсодержащие катализаторы, гидродеароматизация, модельная смесь

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МОДЕЛЬДІК ҚОСПАНЫ ДЕАРОМАТИЗАЦИЯЛАУ ПРОЦЕСІНДЕ МЕЗОКЕУЕКТІ АЛЮМОСИЛИКАТ НЕГІЗІНДЕГІ КАТАЛИЗАТОРЛАРДЫ ЗЕРТТЕУ: ФИЗИКА-ХИМИЯЛЫҚ СИПАТТАМАЛАРЫ ЖӘНЕ КАТАЛИТИКАЛЫҚ БЕЛСЕНДІЛІГІ

Ароматты көмірсутектер мен күкірттің құрамын шектеу Евро-5 экологиялық стандарттарының талаптарына байланысты. Жану кезінде бұл қосылыстар атмосфераға адам денсаулығы мен қоршаған ортаға теріс әсер ететін өте улы қосылыстар шығарады. Сонымен қатар, дизельдегі ароматты көмірсутектердің жоғары концентрациясы оның сапасын едәуір төмендетеді, нәтижесінде күйе пайда болады және цетан саны азаяды. Евро-5 стандарттарына сәйкес полициклді хош иісті көмірсутектер мен күкірт қосылыстарының мөлшері сәйкесінше 8% және 10 мг/кг аспауы керек. Жоғары сапалы дизель отынын өндіру мәселесін шешу үшін гетерогенді катализаторлардың қатысуымен гидротазалау процесі қолданылады.

Мақалада модельдік қоспадағы гидротазалау процесінің белсенділігін зерттеу үшін мезокеуекті алюмосиликаттар мен олардың негізінде бифункционалды катализаторлардың синтезі келтірілген. Алынған үлгілер СЭМ, төмен температурада азоттың адсорбциясы/десорбциясы, рентгендік құрылымдық талдау және инфрақызыл спектрдің Фурье түрленуі сияқты әртүрлі физика-химиялық әдістерді қолдану арқылы зерттелді. Ni/MAS- H-бентонит және Mo/MAS-H-бентонит катализаторлары оңтайлы жағдайларда (240°C, 3 МПа және 0,5 с⁻¹) гидротазалау процесінде жоғары белсенділік пен селективтілік көрсететіні анықталды.

Түйін сөздер: мезокеуекті алюмосиликаттар, бифункционалды катализаторлар, никель-молибден құрамды катализаторлар, гидродеароматизация, модельді қоспа.

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ЗАҚЫМДАНҒАН «КАНАДСКАЯ» СҰРЫПТЫ БИДАЙ ТҰҚЫМЫНАН АЛЫНҒАН СО₂-СЫҒЫНДЫСЫНЫҢ ФИТОХИМИЯЛЫҚ САРАПТАМАСЫ

Аңдатпа: Бұл мақалада «Канадская» сортының тұтас және өнген (зақымданған) бидай тұқымына сапалық және сандық талдау нәтижелері келтірілген. Өнген (зақымданған) бидай дәнінен жоғарыкритикалық флюидті СО₂-экстракциясы арқылы сығынды алынды. Фитохимиялық талдау нәтижесі бойынша СО₂-сығындының құрамында көмірсулар, аминқышқылдар, флавоноидтар, май қышқылдары айқындалды. Газды хроматография әдісі арқылы сығындыда 14 май қышқылы бар екені анықталды, оның басым бөлігі метил миристат (42,57%), метил деканоат (25,85%), цис-10-пентадекен қышқылының метил эфирі (16,80%) және метил палмитат (7,35%) құрады. «Уралосибирская 2» бидай таза тұтас дәнінен алынған СО₂-сығындысымен салыстырғанда зақымдаған «Канадская» бидай сұрыпынан алынған сығындыда айтарлықтай айырмашылық байқалмады. Тек метил лаурат мөлшері 4 есе, метил стеарат – 12 есе, цис-10-гептадецен қышқылының метил эфирі – 3 есе аз болды. Керісінше, зақымдаған бидайдан алынған СО₂-сығындысында метил деканоат мөлшері 4,48 %-ға, ал цис-10-пентадецен қышқылының метил эфирі – 14,52%-ға жоғары болды. Сонымен қатар, осы сығындыда «Уралосибирская 2» бидай таза тұтас дәнінен алынған СО₂-сығындысында болмаған метил ундеканат, метил пальмитат, метил бегенат қаныққан май қышқылдары анықталды. Тағы ескеретін жайт, зақымданған «Канадская» бидай сұрыпынан алынған СО₂-экстракта қанықпаған май қышқылдарының өкілдері идентификацияланды. Жалпы олардың үлесі 19,7% құрады. Капиллярлы электрофорез нәтижелері бойынша В тобындағы дәрумендер ішінен тиаминнің (В1 дәруменінің) үлесі жоғары болды және $0,13 \pm 0,03$ мг/100 г құрады, алайда оның бидай тұқымында болатын