Effect of dibenzothiophene on the hydrogenation of aromatic hydrocarbons over mesoporous materials

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This study investigates the effect of dibenzothiophene on the hydrogenation of aromatic hydrocarbons over bifunctional catalysts based on mesoporous Al-HMS aluminosilicates. Mesoporous aluminosilicate materials with different Si/Al ratios varying from 10 to 70 were synthesized and employed as supports for Ni-Mo-Al-HMS-H-bentonite catalysts. The physico-chemical features of the materials were characterized using ICP-OES, nitrogen physisorption at low temperatures, XRD, FTIR, and pyridine adsorption FTIR spectroscopy. Catalytic activity was evaluated in the hydrogenation of a model mixture of 2-methylnaphthalene and n-hexadecane at temperatures between 220-300°C and at 6 MPa H_2. The optimal catalytic performance was observed for the sample with a Si/Al ratio of 10, displaying high conversion and selectivity to 2-methyldecalin as a result of sufficient Brønsted and Lewis acidity. The presence of dibenzothiophene leads to a decrease in conversion and selectivity due to sulfur-induced catalyst poisoning and shifts the optimal reaction temperature from 240 to 260°C. These findings highlight the important role of acid properties of mesoporous aluminosilicates in hydrogenation and hydrotreatment processes of aromatic compounds.

Keywords: mesopores; aluminosilicates; Si/Al; aromatic compounds; catalyst.

Мезокеуекті материалдарды қолдану арқылы ароматты көмірсутектерді гидрлеу процесіне дибензотиофеннің әсері

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Бұл жұмыста мезокеуекті Al-HMS алюмосиликаттарына негізделген бифункционалды катализаторлардың қатысуымен ароматты көмірсутектердің гидрленуіне дибензотиофеннің әсері зерттеледі. Әртүрлі 10-нан 70-ке дейінгі әртүрлі Si/Al қатынасы бар мезокеуекті алюмосиликат материалдары синтезделіп, Ni-Mo-Al-HMS-H-бентонит катализаторлары үшін тасымалдағыш ретінде пайдаланылды. Алынған материалдардың физика-химиялық қасиеттері ICP-OES, төмен температуралы азот физикалық адсорбциясы, рентген дифракциясы, FTIR спектроскопиясы және адсорбцияланған пиридиннің FTIR спектроскопиясы арқылы сипатталды. Каталитикалық белсенділік 2-метилнафталин мен н-гексадеканның модельді қоспасының 220-300°C температура диапазонында 6 МПа H_2 қысымында гидрлеу реакциясында бағаланды. Бренстед және Льюис қышқыл орталықтарының оңтайлы мөлшеріне байланысты Si/Al қатынасы 10 болатын катализатор мақсатты өнім, 2-метилдекалин үшін жоғары конверсия мен селективтілікті қамтамасыз ететіні анықталды. Дибензотиофенді қосу катализатордың активті орталықтарының күкіртпен улануына байланысты конверсия мен селективтілікті төмендететіні, сондай-ақ оңтайлы процесс температурасын 240-тан 260°C-қа дейін өзгертетіні көрсетілді. Бұл нәтижелер мезокеуекті алюмосиликаттардың қышқылдық қасиеттерінің ароматты қосылыстарды гидрлеу және гидротазартудағы маңызды рөлін растайды.

Түйін сөздер: мезокеуектер; алюмосиликаттар; Si/Al; ароматты қосылыстар; катализатор.

Влияние дибензотиофена на гидрирование ароматических углеводородов с использованием мезопористых материалов

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В работе исследовано влияние дибензотиофена на процесс гидрирования ароматических углеводородов в присутствии бифункциональных катализаторов на основе мезопористых алюмосиликатов Al-HMS. Были синтезированы мезопористые алюмосиликатные материалы с различными соотношениями Si/Al в диапазоне от 10 до 70 и использованы в качестве носителей для катализаторов Ni-Mo-Al-HMS-H-бентонит. Физико-химические свойства полученных материалов были охарактеризованы методами ICP-OES, низкотемпературной физической адсорбции азота, рентгеновской дифракции, ИК-Фурье-спектроскопии и ИК-Фурье-спектроскопии адсорбированного пиридина. Каталитическую активность оценивали в реакции гидрирования модельной смеси 2-метилнафталина и н-гексадекана в температурном интервале 220-300°C и при 6 МПа H_2. Наилучшие каталитические характеристики были получены для образца с соотношением Si/Al, равным 10, который проявил высокую степень конверсии и селективности по целевому продукту 2-метилдекалину, что обусловлено достаточным количеством кислотных центров Бренстеда и Льюиса. Присутствие дибензотиофена приводит к снижению конверсии и селективности вследствие серного отравления катализатора и смещает оптимальную температуру реакции с 240 до 260°C. Полученные результаты подчеркивают важную роль кислотных свойств мезопористых алюмосиликатов в процессах гидрирования и гидроочистки ароматических соединений.

Ключевые слова: мезопоры; алюмосиликаты; Si/Al; ароматические соединения; катализатор.

(2026) Chem Bull Kaz Nat Univ 2:N-NN

CHEMICAL BULLETIN
of Kazakh National University
http://bulletin.chemistry.kz/

IRSTI 61.51.17 https://doi.org/10.15328/cb2026_121

Article

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1. Introduction

The progressive strengthening of environmental requirements for diesel fuel composition stimulates the development of more selective and deep purification processes. Aromatic hydrocarbons, as well as sulfur- and nitrogen-containing compounds, remain among the key factors limiting the production of environmentally compliant fuels. In the United States, regulatory measures lowered the sulfur content in diesel fuel to 15 ppm by 2006 [1]. The Euro 7 framework limits a maximum sulfur level of 10 ppm, with nitrogen oxide emissions regulated within the range of 0.01-0.03 g km^{-1} [2].

Hydrodearomatization (HDA) and hydrodesulfurization (HDS) represent the principal processes employed to decrease the aromatic hydrocarbon content in diesel fuels. It should be noted that fuel dearomatization is often accompanied by the simultaneous occurrence of hydrodesulfurization reactions, since many sulfur-containing components of diesel fractions belong to polyaromatic compounds [3]. The implementation of these processes contributes to the achievement of the United Nations Sustainable Development Goals 3 “Good Health and Well-Being” and 13 “Climate Action” [4].

Due to their high catalytic activity, selectivity, and thermal stability, zeolite catalysts are widely used in deep hydrocarbon processing. In contrast to conventional supports such as γ-Al_2O_3, zeolites exhibit a pronounced shape selectivity effect that promotes directed reaction pathways and enhances selectivity toward target products [5]. However, the microporous nature of zeolites leads to limited accessibility of active sites and diffusion constraints for reactants, which in some cases may reduce their catalytic efficiency [6].

Mesoporous aluminosilicates with an ordered porous structure represent a promising alternative to zeolite catalysts. Their well-developed mesopore system and high specific surface area ensure efficient reactant transport, high dispersion of active sites, and reduced mass transfer limitations, which is particularly important for reactions involving bulky molecules [7].

In HDA and HDS processes, acid sites of moderate strength play a key role. Such sites enable the activation of hydrocarbon compounds without promoting undesirable side reactions, including cracking and coke formation [8-10]. The acidic properties of mesoporous aluminosilicates, including the concentration and strength of acid sites, can be purposefully tuned by varying the Si/Al ratio, which allows optimization of the catalytic performance of the materials in HDA and HDS reactions.

Thus, the aim of the present work is the synthesis of mesoporous aluminosilicate materials with different Si/Al ratios and the preparation of catalysts based on these materials, as well as the investigation of dibenzothiophene effects on aromatic hydrocarbon hydrogenation over the obtained catalytic systems.

2. Experiment

2.1 Materials

All chemicals used were purchased from Sigma-Aldrich and were used without further purification: aluminum-tri-sec-butoxide (aluminum sec-butoxide, 97%, 246.32 g/mol), tetraethoxysilane (TEOS, 98%, 208.33 g/mol), nickel nitrate hexahydrate $Ni(NO_3)_2 \cdot 6H_2O$, 99.9%, 290.79 g/mol), ammonium heptamolybdate tetrahydrate $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$, extra pure,

Received 21 Feb 2026; Received in revised form 14 Apr 2026; Accepted 16 Apr 2026; Available online 30 Jun 2026.

1235.86 g/mol), dibenzothiophene (DBT, 98%, 184.26 g/mol), 2-methylnaphthalene (95%, 142.20 g/mol), n-hexadecane (99%, 226.44 g/mol) and activated bentonite from Tagan field (East Kazakhstan region).

2.2 Preparation of mesoporous aluminosilicate supports and catalysts based on them

The synthesis of mesoporous aluminosilicate materials with different Si/Al ratios and catalysts based on these materials was carried out following the procedure described in [11]. Aluminum sec-butoxide, tetraethyl orthosilicate (TEOS), nickel nitrate hexahydrate, and ammonium heptamolybdate tetrahydrate were used as precursor reagents. Activated bentonite was employed as a secondary support, where the mesoporous aluminosilicate and H-bentonite contents were 35 wt% and 65 wt%, respectively.

2.3 Physico-chemical characterization of the synthesized samples

The Si/Al ratios of the mesoporous aluminosilicates were quantified by inductively coupled plasma optical emission spectroscopy (ICP-OES) using a Spectro Arcos Multiview FHX22 instrument. Structural ordering was evaluated by X-ray diffraction (XRD) on a Philips X'PERT MPD diffractometer employing Cu Kα radiation (λ = 0.15418 nm). Textural characteristics were determined from nitrogen adsorption-desorption isotherms measured at 77 K with a Micromeritics Tristar 3000 analyzer. The specific surface area was calculated according to the Brunauer-Emmett-Teller (BET) method, whereas pore volume and pore size distribution were obtained using the Barrett-Joyner-Halenda (BJH) approach. Fourier-transform infrared (FTIR) spectra were collected on a Thermo Scientific Nicolet iS50 spectrometer,and the acidic properties of the materials were investigated by pyridine adsorption followed by FTIR analysis on a Jasco FT/IR-4600 instrument.

2.4 Evaluation of the catalytic performance of bifunctional catalysts

Catalytic performance tests were conducted in a high-pressure reactor at temperatures of 220-300°C and at 6 MPa H_2, with a reaction duration of 5 hours. A model mixture consisting of 9 wt% 2-methylnaphthalene, 300 ppm DBT, and 91 wt% n-hexadecane was used to simulate the principal components of diesel fuel. The catalyst loading was 0.2 g, corresponding to a catalyst/feedstock ratio of 1:50. Prior to catalytic testing, the catalyst was sulfided in a flow reactor at 400°C for 5 h in a H_2 stream containing 10 vol% H_2S, with a heating rate of 4°C/min. The active Ni-Mo phase is generally considered to consist of Ni^{2+} and Mo^{4+} species. The liquid reaction products were characterized by GC-MS using a Shimadzu GCMS-QP2010 fitted with a Restek Rxi-5 ms capillary column. All catalytic experiments were performed in triplicate.

3. Results and Discussion

The porous structure of Al-HMS mesoporous aluminosilicates was examined by low-temperature nitrogen adsorption-desorption. The main textural properties of the samples with Si/Al ratios of 10, 30, 50, and 70 are summarized in Table 1.

Table 1 – Influence of the Si/Al ratio on the textural characteristics of Al-HMS

Sample	Initial Si/Al ratio	Si/Al*	BET area, m^2/g	Pore volume, cm^3/g	Average pore diameter, nm
Al-HMS	10	9	818	0.87	3.47
	30	29	901	0.96	3.49
	50	50	932	0.96	3.86
	70	68	956	0.98	3.86

* Si/Al ratio determined by ICP-OES

As shown in Table 1, all synthesized Al-HMS materials with Si/Al ratios of 10, 30, 50, and 70 display a well-developed mesoporous structure. This is evidenced by high specific surface

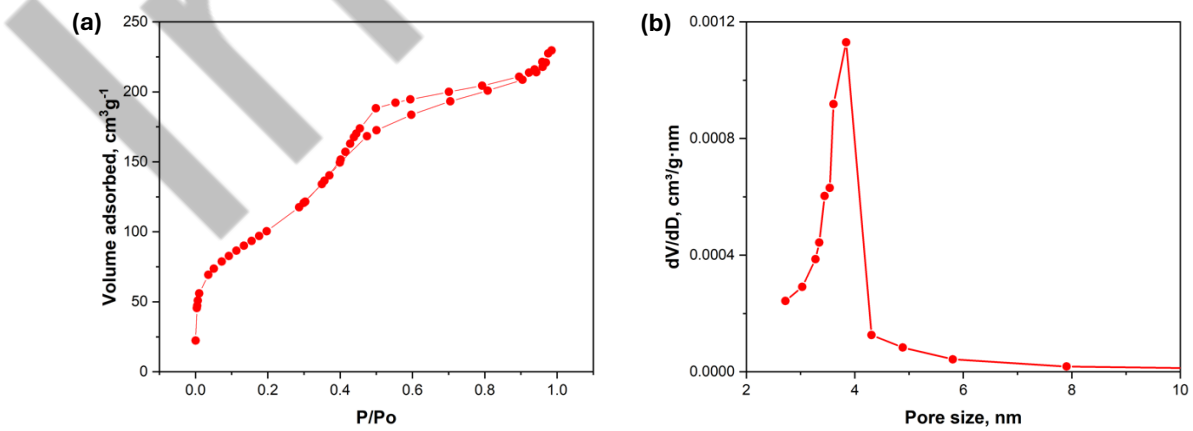

Figure 1 – Nitrogen physisorption isotherm (a) and pore size distribution (b) for Al-HMS(10)

areas (818–956 m^2/g), pore volumes (0.87–0.98 cm^3/g), and average pore diameters (3.47-3.86 nm). A decrease in aluminum content leads to an improvement in the textural properties of the synthesized aluminosilicates, which may be attributed to reduced framework distortion at lower aluminum content [12, 13].

Figure 1 displays nitrogen physisorption isotherm (a) and pore size distribution (b) of Al-HMS(10) as a representative example.

The nitrogen physisorption isotherm shown in Figure 1a correspond to type IV with an H4 hysteresis loop classified according to IUPAC standards. The appearance of hysteresis at relative pressures $P/P_0 > 0.4$ is associated with capillary condensation of nitrogen within the mesoporous structure [14]. The average pore diameter determined from the pore size distribution curves is 3.47 nm (Figure 1b).

X-ray diffraction measurements provided additional insight into the structural formation of Al-HMS-based catalysts with varying Si/Al ratios. The XRD patterns of Al-HMS(10) and Ni-Mo-Al-HMS-H-bentonite catalysts recorded in the 2θ range of 10-90° are shown in Figure 2.

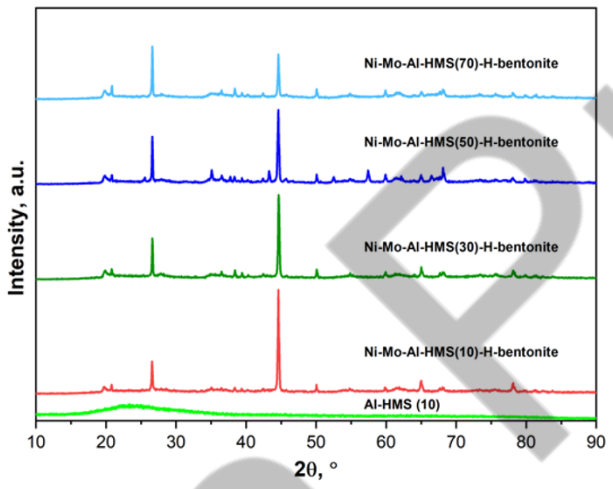

Figure 2 – XRD patterns of Al-HMS(10) and Al-HMS based catalysts with different Si/Al ratios

The resulting diffraction patterns indicate the presence of crystalline Ni and Mo phases in the catalyst composition. In the 2θ range of 20-30°, Al-HMS(10) exhibits the amorphous nature of the silica matrix (Figure 2). The diffraction peak at 2θ = 27.3° corresponds to the MoO_3 phase, whereas the peaks at 44.6 and 62.7° can be attributed to the cubic NiO phase [15–17].

In the case of Ni-Mo-Al-HMS(50)-H-bentonite, additional reflections are observed. A reflection around 26° can be associated with $NiMoO_4$ [18], whereas the peaks at 35.3°, 53.1°, and 58.3° are assigned to MoO_3 [19, 20], and those at 43.3° and 66.1° to NiO [21, 22]. These reflections are observed exclusively for this sample, while in the other samples these reflections are either absent or significantly less intense, suggesting a lower extent of crystallization of the corresponding oxide phases.

Also, a gradual decrease in the intensity of diffraction peaks is observed with increasing Si/Al ratio from 10 to 70, indicating a reduction in the crystallinity of the oxide phases. This trend suggests that higher Si/Al ratios promote a more uniform distribution of metal species and suppress the formation of well-defined crystalline domains [23, 24].

To investigate the interaction between silicon and aluminum within the framework and the presence of hydroxyl groups, Fourier transform infrared (FTIR) spectra of Ni-Mo-Al-HMS-H-bentonite catalysts based on Al-HMS materials with varying Si/Al ratios were recorded. The corresponding spectra are shown in Figure 3.

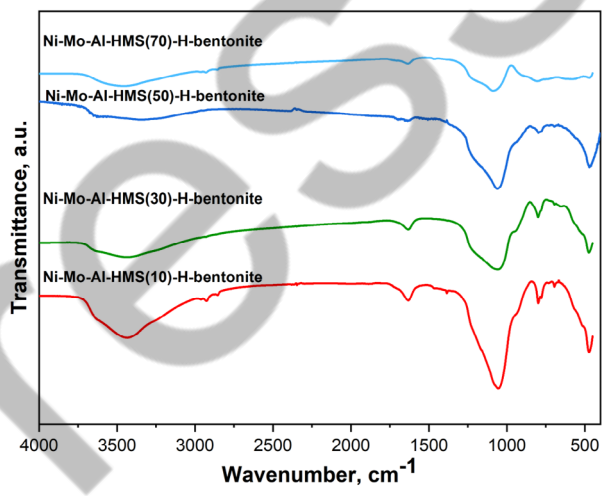

Figure 3 – FTIR spectra of bifunctional Al-HMS catalysts with different Si/Al ratios

All FTIR spectra show a broad absorption feature in the 3700-3300 cm^{-1} region (Figure 3), attributed to hydroxyl group vibrations and hydrogen-bonded water molecules. The higher intensity of this band observed for the Ni-Mo-Al-HMS(10)-H-bentonite and Ni-Mo-Al-HMS(30)-H-bentonite catalysts may be attributed to the presence of hydroxyl groups related to extra-framework aluminum species [25]. The absorption band at 1635 cm^{-1} is attributed to O-H bending vibrations. Increasing the Si/Al ratio results in a reduced intensity of hydroxyl-related bands, which reflects an enhanced degree of silanol condensation. The bands at 1064, 800, and 472 cm^{-1} correspond to Si-O-Si, Al-O-Al, and Al-O-Si vibrational modes characteristic of the aluminosilicate framework, respectively [26, 27].

The acidic properties of the synthesized catalysts were investigated by diffuse reflectance infrared Fourier transform spectroscopy using adsorbed pyridine (Py-FTIR). The Py-FTIR spectra of catalysts with different Si/Al ratios are shown in Figure 4.

Figure 4 reveals that the absorption feature at 1545 cm^{-1} is characteristic of Brønsted acid sites, while the band near 1455 cm^{-1} is indicative of Lewis acidity. The signal detected at 1490 cm^{-1} arises from contributions of both Brønsted and Lewis acid sites [28]. With increasing aluminum content, the intensities of these bands become more pronounced, reflecting a higher concentration of both types of acid sites.

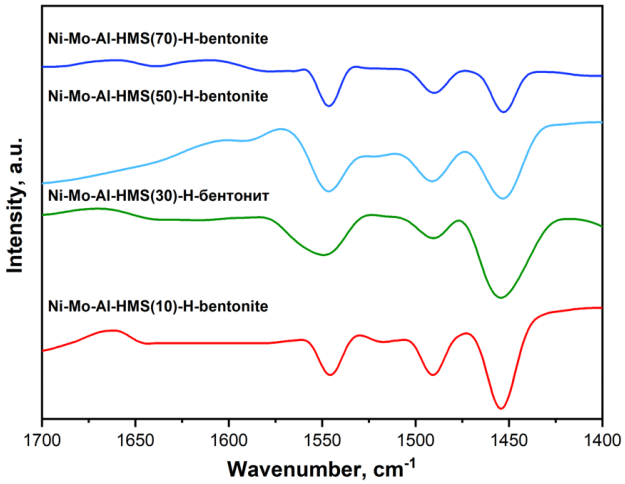

Figure 4 – Py-FTIR spectra of bifunctional Al-HMS catalysts with different Si/Al ratios

The catalytic activity of the obtained bifunctional catalysts was first investigated in the hydrotreatment of a model mixture of 2-methylnaphthalene and n-hexadecane in the absence of sulfur-containing compounds. Figure 5 shows the dependence of 2-methyldecalin (2MD) selectivity on the different Si/Al ratio in Ni-Mo-Al-HMS-H-bentonite catalysts.

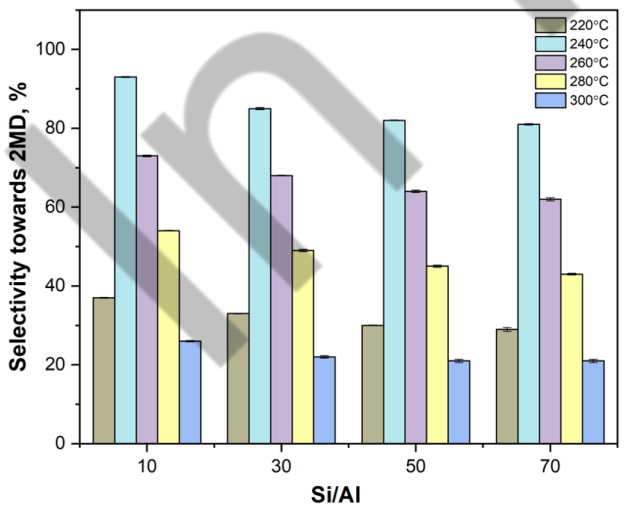

Figure 5 – Dependence of 2MD selectivity on the Si/Al ratio in Ni-Mo-Al-HMS-H-bentonite catalysts

A maximum selectivity of 93% toward the target product (2MD) is achieved over Al-HMS containing a Si/Al ratio of 10 at a reaction temperature of 240°C (Figure 5). Higher Si/Al ratios lead to a lower concentration of Brønsted and Lewis acid sites, thereby decreasing selectivity and hydrogenation efficiency [29-32].

Among the investigated materials, Al-HMS (Si/Al = 10) demonstrated the highest hydrogenation efficiency and was selected for further investigation. DBT was additionally introduced into the model reaction system in order to investigate the effect of a sulfur-containing compound on the hydrogenation process.

Table 2 summarizes the results of the study on the effect of DBT on the hydrogenation of a 2-methylnaphthalene and n-hexadecane mixture over the Ni-Mo-Al-HMS-H-bentonite catalyst at temperatures of 220-300°C, at 6 MPa H_2 and a reaction time of 5 hours.

Table 2 – Influence of DBT on 2-methylnaphthalene hydrogenation over Ni-Mo-Al-HMS(10)-H-bentonite

Parameter	Temperature, °C				
	220	240	260	280	300
Conversion, %	89±0.3	91±0.2	92±0.4	92±0.1	95±0.5
Selectivity, %					
2MD	31±0.2	21±0.3	68±0.4	49±0.1	41±0.4
2MT	14±0.2	36±0.4	13±0.1	24±0.1	22±0.3
6MT	55±0.4	43±0.1	19±0.3	27±0.3	37±0.3

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

Figure 6 compares the catalyst selectivity toward reaction products in hydrogenation experiments performed with and without DBT.

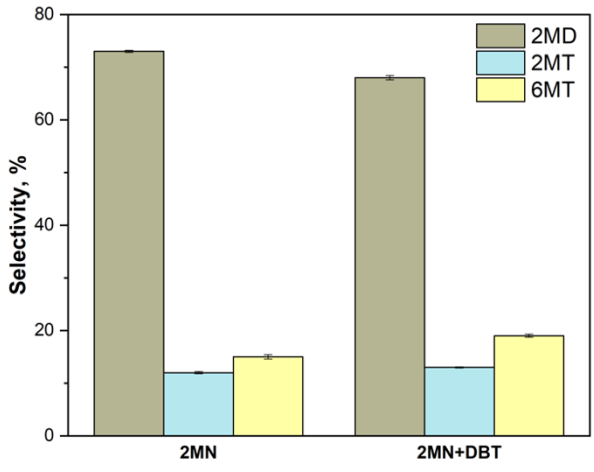

Figure 6 – Effect of DBT on 2-methylnaphthalene hydrogenation selectivity at 260°C over Ni-Mo-Al-HMS(10)-H-bentonite

At a reaction temperature of 260°C, the conversion of 2-methylnaphthalene reaches 98% in the absence of sulfur-containing compounds, whereas the addition of dibenzothiophene results in a decrease in conversion to 92%, corresponding to a reduction of 6% (Table 2). The selectivity toward the target product, 2-methyldecalin, attains 73% at 260°C in the sulfur-free system but decreases to 68% in the presence of dibenzothiophene, representing a 5% reduction (Figure 6). The observed decrease in conversion and selectivity is attributed to sulfur-induced catalyst poisoning caused by the adsorption of dibenzothiophene molecules on the active sites, which reduces their availability for reactant activation.

Conclusion

Thus, mesoporous aluminosilicate materials with different Si/Al ratios and Ni-Mo-Al-HMS-H-bentonite catalysts based on these materials were successfully synthesized. The physicochemical properties of the obtained materials were investigated using ICP-OES, nitrogen physisorption at low temperatures, XRD, FTIR, and pyridine adsorption FTIR spectroscopy. The catalytic activity of the samples was evaluated in the hydrogenation of a 2-methylnaphthalene-n-hexadecane mixture, and the effect of dibenzothiophene on the process efficiency was also assessed. It was established that, in the hydrogenation of 2-methylnaphthalene, the Ni-Mo-Al-HMS(10)-H-bentonite catalyst (Si/Al = 10) provides the maximum conversion and selectivity toward the target product. The high catalytic performance of this sample is attributed to the presence of both Brønsted and Lewis acid sites. The optimal reaction temperature for the sulfur-free model mixture is 240°C. The addition of dibenzothiophene increases the optimal process temperature to 260°C, which is associated with additional energy requirements in the presence of sulfur.

CRediT authorship contribution statement

A. Abdrassilova: Writing – review & editing, Writing – original draft, Validation, Supervision, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. *G. Vassilina:* Visualization, Validation, Formal analysis. *K. Abdildina:* Investigation, Data curation, Software. *T. Abildin:* Investigation, Data curation.

Conflicts of Interest

The authors report no competing financial interests or personal relationships that could have influenced the research presented in this work.

Acknowledgements

This research was funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan AP32724827.

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