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SYNTHESIS OF A BIFUNCTIONAL CATALYST BASED ON REGENERATED Al_2O_3 ADSORBENT

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Abstract

To synthesize a bifunctional catalyst on an Al_2O_3 support, the initially used adsorbent Al_2O_3 was regenerated at 600 °C for 360 minutes in the absence of atmospheric oxygen. A process model for synthesizing bifunctional catalysts on regenerated Al_2O_3 support was developed using Ni, Co, Mo, and P precursors. According to the elemental composition analysis of the regenerated adsorbent by XRF (X-ray fluorescence) method, it was determined to consist of 81.5 % Al_2O_3. Using the regenerated Al_2O_3 as the support, $(NH_4)_6Mo_7O_{24}\cdot4H_2O$, Ni A process model was developed to synthesize a bifunctional catalyst using salts such as $Ni(NO_3)_2\cdot6H_2O$, $Co(NO_3)_2\cdot6H_2O$, and H_3PO_4 acid, and based on this model, three types of catalyst samples were synthesized. To determine the morphological changes and textural properties of the surface of the obtained catalyst samples, analyses were performed using SEM (Scanning Electron Microscope), TEM (Transmission Electron Microscope), and BET (Brunauer-Emmett-Teller) model-based methods. These analyses were applied to evaluate morphological changes and textural characteristics of the prepared catalyst samples.

Keywords: regeneration; carrier; catalyst; hydrotreating; aluminum oxide; synthesis; textural properties.

СИНТЕЗ БІФУНКЦІОНАЛЬНОГО КАТАЛІЗАТОРА НА ОСНОВІ РЕГЕНЕРОВАНОГО АДСОРБЕНТУ Al_2O_3

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Анотація

Для синтезу біфункціонального каталізатора на носії Al_2O_3 попередньо використаний адсорбент Al_2O_3 був регенерований за температури 600 °С протягом 360 хв за відсутності атмосферного кисню. Згідно з результатами аналізу елементного складу регенерованого адсорбенту методом рентгенофлуоресцентної спектроскопії (РФС), було встановлено, що він складається з 81.5 % Al_2O_3. З використанням регенерованого Al_2O_3 як носія, була розроблена технологічна модель синтезу біфункціонального каталізатора із застосуванням солей $(NH_4)_6Mo_7O_{24}\cdot4H_2O$, $Ni(NO_3)_2\cdot6H_2O$, $Co(NO_3)_2\cdot6H_2O$ та кислоти H_3PO_4. На основі розробленої моделі були синтезовані три типи зразків каталізаторів. Для визначення морфологічних змін та текстурних властивостей поверхні отриманих зразків каталізаторів були проведені аналізи з використанням скануючої електронної мікроскопії (СЕМ), трансмісійної електронної мікроскопії (ТЕМ) та методів, заснованих на моделі БЕТ (Брунауера-Еммета-Теллера).

Ключові слова: регенерація; носій; каталізатор; гідроочищення; алюміній оксид; синтез; текстурні властивості.

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Introduction

Catalytic processes are a key technological component of the modern oil and gas industry. Reactions carried out in the presence of catalysts allow for achieving high selectivity, high efficiency, and a significant reduction in energy consumption [1–3]. Today, over 80 % of crude oil processing worldwide is carried out using catalytic processes [4–6].

Worldwide, the production volumes of the petrochemical industry and the petrochemical synthesis industry exceed $700 billion. It should also be noted that approximately 200,000 tons of catalysts are produced each year at various plants worldwide, with a total value of about 15–20 billion US dollars [7; 8].

The active development of the global chemical, oil, and gas industries has led to an increased demand for effective adsorbents and catalysts for the purification of petroleum products and the production of targeted, high-quality products. In this regard, the development of effective modified and selective catalysts based on local secondary raw materials and their introduction into the processes of hydrogenation and hydrotreating of petroleum products is currently of particular importance in the field of oil and gas refining and petrochemistry within the country. [9].

Today, the annual global demand for catalysts in oil and gas processing processes amounts to several thousand tons, and about 30 % of that is used in the hydrotreating processes to remove heteroatoms from oil and petroleum products. [10].

In the coming years, Uzbekistan will rank high in oil, gas, and gas condensate production. This will help the republic produce high-quality fuel and establish a raw material base to supply products for the chemical industry in the future. In Uzbekistan, there are numerous natural gas fields and their reserves. This increases the production of gas condensate, which is extracted along with the gas during extraction [11].

However, no matter how advanced the petroleum refining industry and other sectors are, their development is limited by the technical level, which lags behind the world's technical standards in the area of complex product processing, especially high-quality products – gasoline, kerosene, diesel fuel, alcohols, plasticizers, paraffins, additives, and other valuable chemical materials - are lagging behind in production. Along with the introduction of new catalytic processes into industry to solve these issues (catalytic cracking, catalytic reforming, alkylation, polymerization, hydrotreating, etc.), the synthesis and industrial introduction of fundamentally new catalysts is a pressing task Performing two to three processes in one reactor with a multifunctional catalyst helps to overcome the disadvantages mentioned above [12–14].

The main directions for the development of the oil refining process are as follows: improving catalyst activity stability, obtaining high-quality products from raw materials, increasing process selectivity, and improving and optimizing the process parameters in terms of pressure reduction. The development of the petrochemical synthesis industry is dependent on the efficiency and operational stability of catalytic processes and catalysts. However, at present, various catalysts produced on an industrial scale in different aggregate states have been described and distinguished in a non-systematic manner, based only on the following parameters [15]:

a) the type of reaction to be catalyzed, according to the oxidation-reduction and acid-base properties of the catalysts;

b) the nature of the active substance, i.e., metal, sulfide, metal-organic, and complex catalysts;

c) catalytic or technological characteristics, distinguished by the cracking of petroleum products, the synthesis of various substances, and different processes.

Based on the data presented in [16-19], catalytic processes are considered important areas of petroleum refining, chemical, and petrochemical industries, and are described as follows (Table 1).

Currently, a large number of catalysts are being produced on an industrial scale in the oil refining and petrochemical industries. However, most of these catalysts were introduced 20–30 years ago. Catalysts prepared and applied on the basis of new theories are virtually nonexistent [20; 21].

In our country's oil refineries, alumina-cobalt-molybdenum (ACM) or alumina-nickel-molybdenum (ANM) catalysts are widely used in the hydrotreating and hydrogenation processes of fuel products. The main raw material in the production of these types of catalysts is aluminum oxide (Al_2O_3), which serves as the support for virtually all precious and selective catalysts. At oil refineries, catalysts used in the hydrotreating process accumulate in large quantities as waste. This has a negative impact on aquatic ecosystems due to the potential leaching of metal compounds contained in them into water. Therefore, research on recycling and extending the service life of catalysts used in the hydrotreating process is of great importance [22].

Table 1

Main directions for the use of catalytic processes in oil refining

Catalytic Process		**Main Catalyst Types**
Cracking		Zeolite- and aluminosilicate-based microspherical and spherical catalysts containing oxides of alkali and alkaline-earth metals
Hydrogenation processes	**Hydrotreating**	Zeolites modified with Al_2O_3 binders exhibiting hydrogenation functionality (Pt-group metals and Ni, Co, W oxides)
	Desulfurization	
	Hydrocracking	Hydrogenation-active zeolites modified with Al_2O_3 binders (Pt-group metals and Ni, Co, W oxides).
Reforming		Chlorinated and fluorinated Al_2O_3-supported catalysts containing platinum (0.2–0.6 wt.%)
Isomerization		Zeolites supported on zirconia- and fluorinated Al_2O_3 containing platinum

At Bukhara Oil Refining Plant LLC, Al_2O_3 is used as an adsorbent to purify hydrogen-containing gas from hydrogen chloride in the gasoline fraction reforming technological system [23–25]. The used aluminum oxide is not regenerated and has accumulated in large quantities at the plant as waste.

Taking into account the above data and with the aim of using the spent aluminum oxide as a support for obtaining high-quality and selective catalysts in the future, this work set the objective of regenerating the aluminum oxide used in the study and synthesizing a bifunctional catalyst for hydrogenation and hydrotreating processes based on it.

The aim of this study is to regenerate a spent Al_2O_3 adsorbent used in hydrogen-containing gas purification and to develop a bifunctional Ni–Co–Mo–P/Al_2O_3 catalyst based on the regenerated support for hydrotreating and hydrogenation processes.

The objectives of the research are as follows:

– to investigate the physicochemical state of the spent Al_2O_3 adsorbent prior to regeneration, including the presence of carbonaceous deposits;

– to determine the optimal temperature and duration for thermal regeneration of the spent Al_2O_3 in an oxygen-free atmosphere;

– to evaluate changes in the elemental composition of Al_2O_3 before and after regeneration using X-ray fluorescence analysis;

– to develop a sequential model for the synthesis of bifunctional catalysts based on regenerated Al_2O_3;

– to synthesize Ni–Co/Al_2O_3, Ni–Co–Mo/Al_2O_3 and Ni–Co–Mo–P/Al_2O_3 catalysts by the impregnation method;

– to study the morphological and textural properties of the synthesized catalysts using SEM, TEM and N_2 adsorption–desorption (BET) analysis.

Materials and methods

The object of study was spent adsorbent Al_2O_3 used in the gasoline fraction reforming technological system of Bukhara Oil Refinery LLC

In conducting the research, the thermal and steam methods for regenerating aluminum oxide, the “impregnation” [24] method for obtaining a bifunctional catalyst, and modern techniques for analyzing the resulting data were employed. The bifunctional catalysts were synthesized by the incipient wetness impregnation method. Aqueous solutions of $Ni(NO_3)_2·6H_2O$, $Co(NO_3)_2·6H_2O$ and $(NH_4)_6Mo_7O_{24}·4H_2O$ were prepared in calculated concentrations. The regenerated Al_2O_3 support was impregnated stepwise with the metal salt solutions under continuous stirring to ensure uniform distribution of the active components. After impregnation, the samples were dried at 110 °C for 12 h and subsequently subjected to thermal treatment. For phosphorus-modified samples, H_3PO_4 was introduced during the final impregnation step.

The elemental composition of the regenerated Al_2O_3 was determined by X-ray fluorescence analysis (XRF) using a Shimadzu EDX-7000FP instrument with the FP (Fundamental Parameters) method.

The size and dispersion of Ni, Co and Mo particles deposited on the Al_2O_3 support were determined from TEM and HRTEM images using statistical image analysis. The dispersion of the active phase was calculated based on the Kasztelan hexagonal model, which is commonly applied for layered Mo-based catalytic systems. The morphological changes on the surface of the synthesized catalysts were observed by SEM images, and their internal structure, crystal lattice, particle size, and active phase distribution were determined from TEM images using a Tencai G2 20 instrument with a LaB6 cathode at an accelerating voltage of 200 kV. The samples were deposited on copper grids coated with a carbon film. HRTEM images of the samples were taken at a magnification of approximately 200,000 under the bright-field zone under a phase-contrast condition without an objective aperture. The dispersion (D) of the active phase particles was calculated based on the Kasztelan hexagonal model.

The textural properties of the samples of synthesized bifunctional catalysts were determined by obtaining low-temperature N_2 adsorption–desorption isotherms on an Autosorb-1 (Quantachrome) porosimeter and performing BET-model-based analyses of the resulting data.

Results and discussion

Prior to regeneration, the spent Al_2O_3 adsorbent was characterized by visible dark coloration and increased mass, which is typical for alumina materials deactivated by carbonaceous deposits during prolonged industrial use. The presence of coke deposits was indirectly confirmed by subsequent mass loss during thermal treatment, indicating the suitability of thermal regeneration as an effective method for restoring the adsorbent properties.

The spent adsorbent Al_2O_3, used to purify hydrogen-containing gas from hydrogen chloride in the technological system for reforming gasoline fractions at Bukhara Oil Refinery LLC, was subjected to dry thermal treatment in the temperature range of 300–900 °C without the presence of air oxygen, and the results obtained are presented in Table 2.

Table 2

Results of thermal treatment of Al_2O_3, in the dry temperature range of 300-900oC without the presence of atmospheric oxygen

Sample	Temperature, °C	Time, min.	Initial weight of the sample, g	Sample weight after regeneration, , g
A-1	300	360	500	484
A-2	400	360	500	462
A-3	500	360	500	453
A-4	600	360	500	435
A-5	700	360	500	435
A-6	800	360	500	435
A-7	900	360	500	435

As can be seen from the research results presented in Table 2, when thermally treating the adsorbent Al_2O_3 without the presence of air oxygen at temperatures from 300 °C to 600 °C, the adsorbent weight decreased from 484 g to 435 g, respectively. However, when the temperature was further increased to 900 °C, the adsorbent weight remained unchanged. Based on these results, it can be concluded that during the thermal regeneration of the used adsorbent Al_2O_3 in an oxygen-free atmosphere, the adsorbent loses a maximum amount of weight at 600 °C and the regeneration degree reaches its maximum [26].

A comparative analysis of the Al_2O_3 adsorbent before and after thermal regeneration indicates significant changes in its physicochemical composition. Prior to regeneration, the spent Al_2O_3 contained carbonaceous deposits formed during prolonged industrial operation, which partially blocked the pore structure and reduced the relative content of active alumina. After thermal treatment at 600 °C for 360 minutes in an oxygen-free atmosphere, these deposits were effectively removed, leading to a noticeable decrease in sample mass and an increase in the relative Al_2O_3 content. XRF analysis of the regenerated material confirmed that Al_2O_3 became the dominant phase, accounting for 81.5 wt.%, which demonstrates the effectiveness of the regeneration process and the suitability of the treated adsorbent for use as a catalyst support.

Based on the research results obtained from the thermal regeneration of the used Al_2O_3 adsorbent, a graph of the adsorbent weight versus regeneration temperature was developed, and this graph is presented in Figure 1.

Taking into account that the regeneration degree of the used Al_2O_3 reaches its maximum at 600 °C, studies were also carried out to determine the time dependence of the regeneration process. The obtained results are presented in Table 3 and Figure 2.

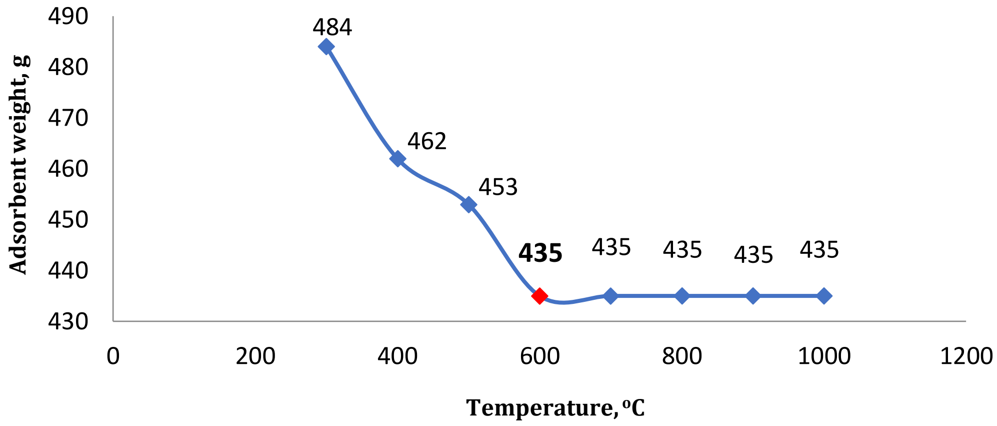


Fig. 1. Graph of the dependence of adsorbent weight on regeneration temperature during thermal regeneration of Al_2O_3

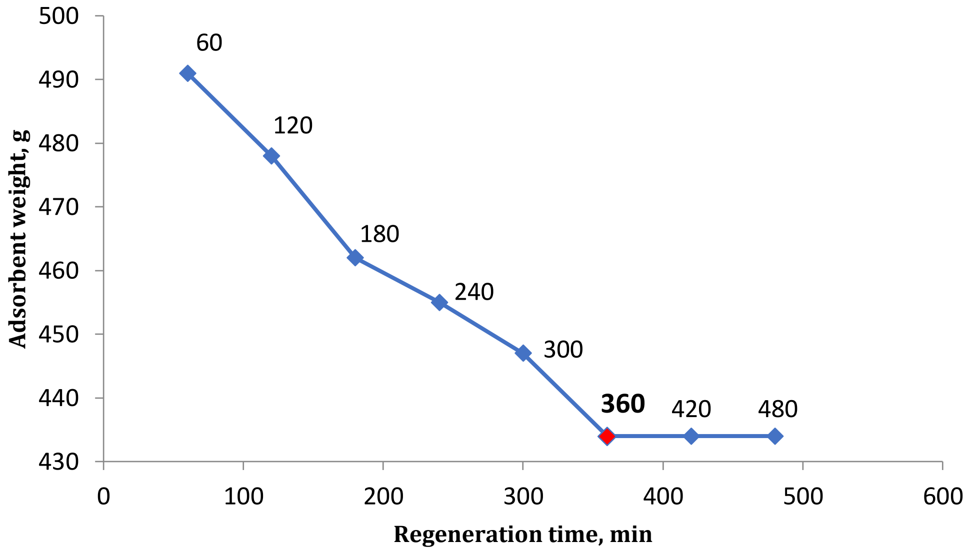


Fig. 2. Time dependence of the regenerating temperature of the adsorbent mass at a constant 600 °C

As shown in Table 3 and the nomogram in Figure 2, the process time for thermal regeneration of Al_2O_3 at a constant temperature of 600 °C ranges from 60 to 360 min. As the process time varied in the 60–360 min range, the adsorbent weight correspondingly decreased from 491 to 434 g; however, when the time was further increased to 360–480 min, the adsorbent weight remained unchanged.

Table 3

Results of thermal treatment of Al_2O_3 at 600°C without the presence of air oxygen

Sample	Temperature, °C	Time, min.	Initial weight of the sample, g	Sample weight after regeneration, g
A-1	600	60	500	491
A-2	600	120	500	478
A-3	600	180	500	462
A-4	600	240	500	455
A-5	600	300	500	447
A-6	600	360	500	434
A-7	600	420	500	434
A-8	600	480	500	434

Figure 3 shows the initial (a) and regenerated (b) appearances of the Al_2O_3 used.

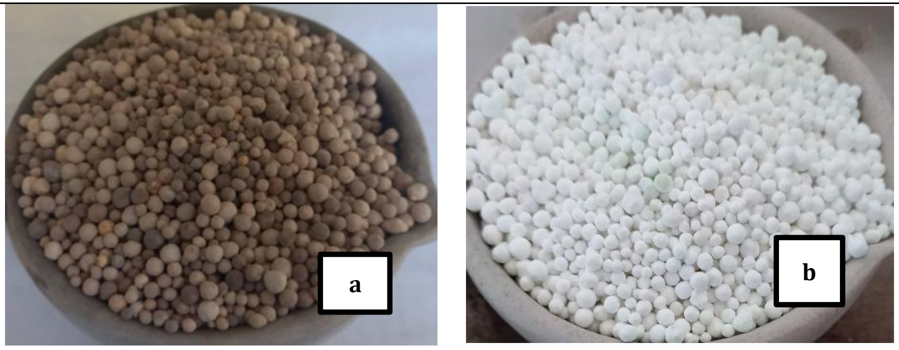
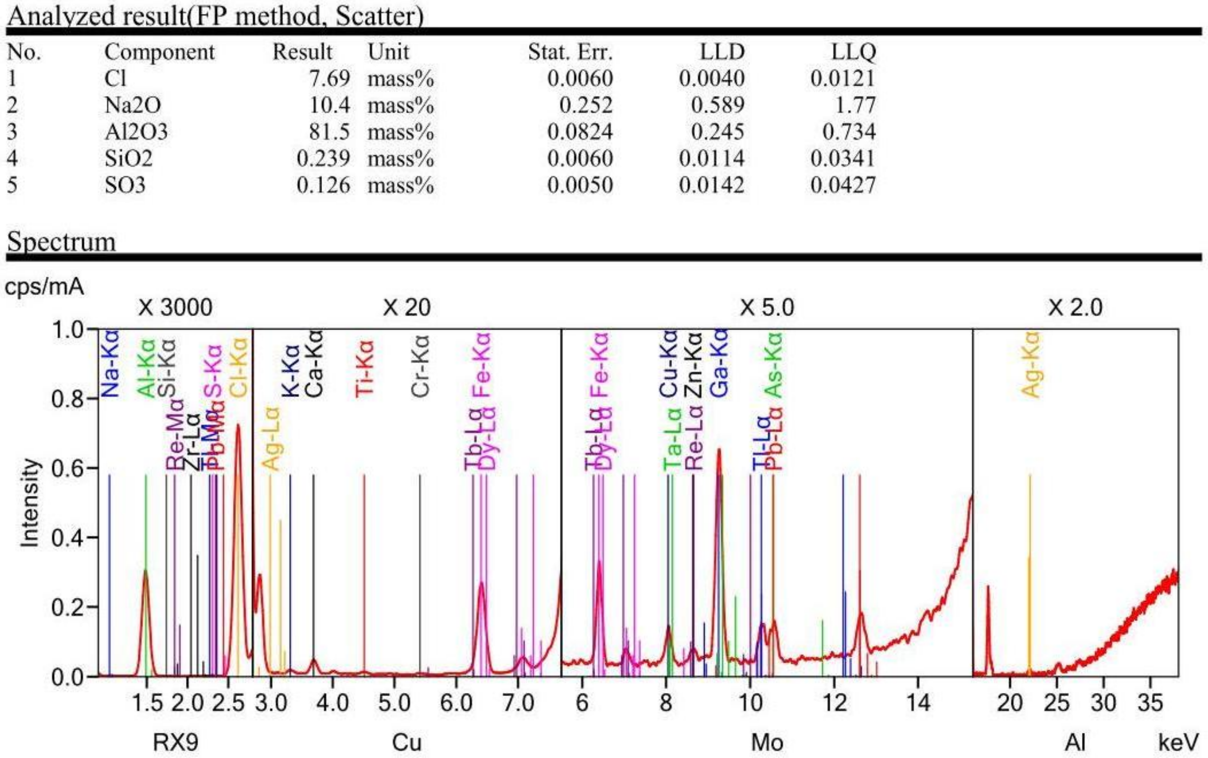


Fig. 3. Initial (a) and regenerated (b) appearance of the used Al_2O_3.

Based on the results of studies on the thermal regeneration of the used adsorbent Al_2O_3, the optimal conditions for the regeneration process were determined to be a temperature of 600 °C and a process duration of 360 minutes.

The elemental composition of the regenerated Al_2O_3 was determined by X-ray fluorescence analysis (XRF) using the Shimadzu EDX-7000FP instrument with the FP (Fundamental Parameters) method. The obtained results are presented in Figure 4.

As shown in Figure 4, the XRF analysis results indicate that the main component of the thermally regenerated adsorbent is active Al_2O_3 (81.5 %). Therefore, this adsorbent was proposed as a carrier for synthesizing bifunctional catalysts for the hydrogenation and hydrodeactivation processes. A sequential process model for synthesizing bifunctional catalysts based on regenerated adsorbent Al_2O_3 was developed (Figure 5).



Analyzed result(FP method, Scatter)

No.	Component	Result	Unit	Stat. Err.	LLD	LLQ
1	Cl	7.69	mass%	0.0060	0.0040	0.0121
2	Na2O	10.4	mass%	0.252	0.589	1.77
3	Al2O3	81.5	mass%	0.0824	0.245	0.734
4	SiO2	0.239	mass%	0.0060	0.0114	0.0341
5	SO3	0.126	mass%	0.0050	0.0142	0.0427

Spectrum

Fig. 4. XRF analysis spectrogram of the regenerated Al_2O_3

Based on the obtained XRF results, a sequential process model for the synthesis of bifunctional catalysts using regenerated Al_2O_3 as a support was developed (Figure 5).

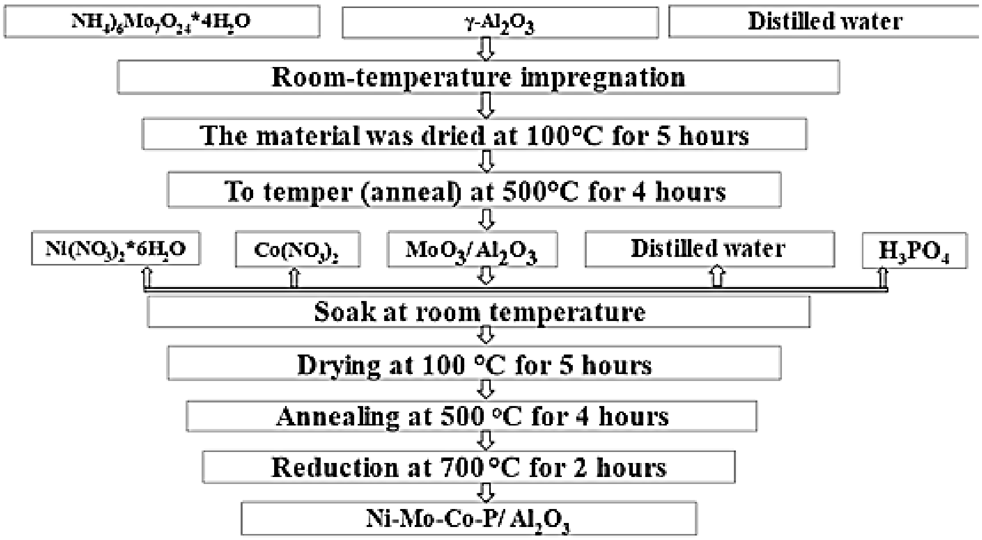


Fig. 5. Model for synthesizing a bifunctional catalyst based on regenerated Al_2O_3

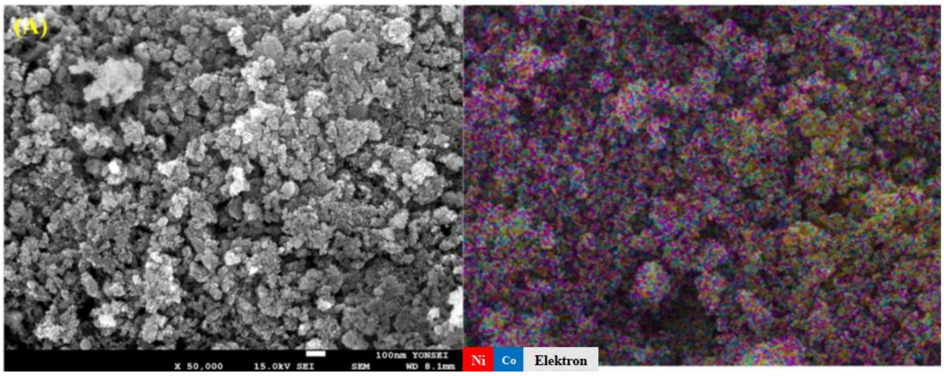
Following the sequence of processes in this model, three types of catalysts, as shown in Figure 6, were synthesized.

a b c

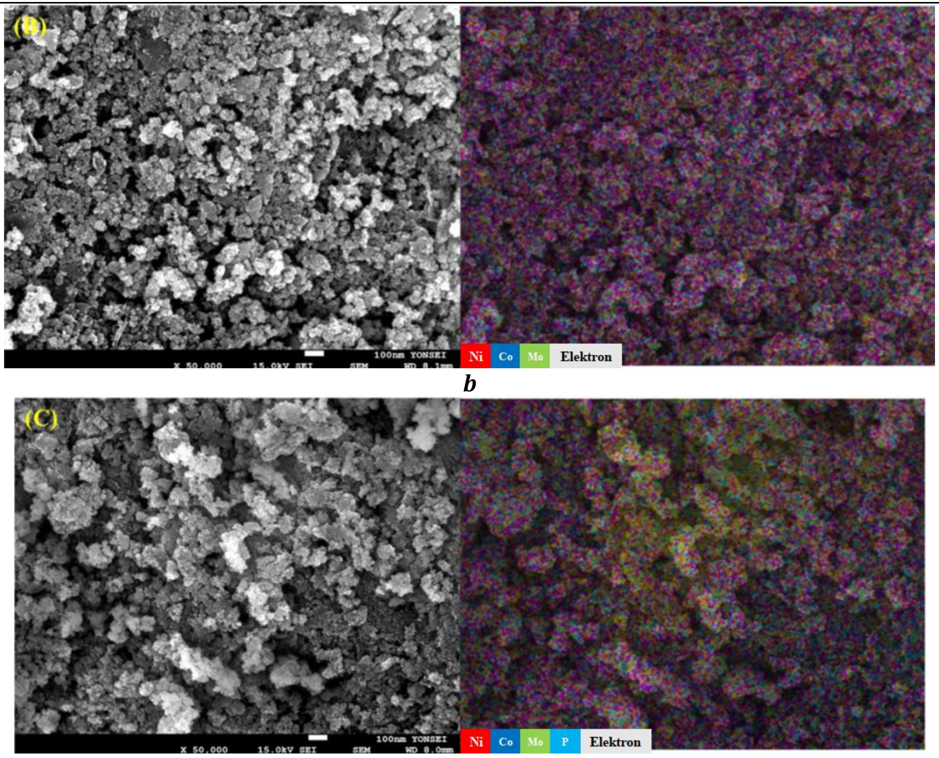
Fig. 6. Samples of bifunctional catalysts prepared on regenerated Al_2O_3: a – Ni–Co/ Al_2O_3, b – Ni–Co–Mo/ Al_2O_3, c – Ni–Co–Mo–P/ Al_2O_3

To study the adsorption of Ni, Co, Mo, and P active components onto the carrier Al_2O_3 and their effect on surface-morphological changes, SEM analyses of the bifunctional catalyst samples prepared on regenerated Al_2O_3 were carried out. SEM images are shown in Figure 6.

Analysis of the SEM images revealed that the active components Ni, Co, Mo, and P were adsorbed onto the Al_2O_3 carrier and uniformly distributed on its surface. It was determined that the impregnation of the active components onto the Al_2O_3 carrier significantly increased their dispersion, which enhanced the initial activity and stability, and due to the agglomeration of the active particles and the Al_2O_3 carrier, larger, clearly bound Al_2O_3, dense-like structure was observed for the Al_2O_3, due to the agglomeration of the active particles and the Al_2O_3 carrier.



a



b

c

Fig. 6. SEM images of catalyst samples: a – Ni-Co/Al_2O_3, b – Ni-Co-Mo/Al_2O_3, c – Ni-Co-Mo-P/Al_2O_3

High-resolution TEM analyses were performed on the synthesized catalysts to determine their internal structure, crystal lattice, and the size and distribution of the active phase particles. The results obtained are presented in Figures 7 and 8.

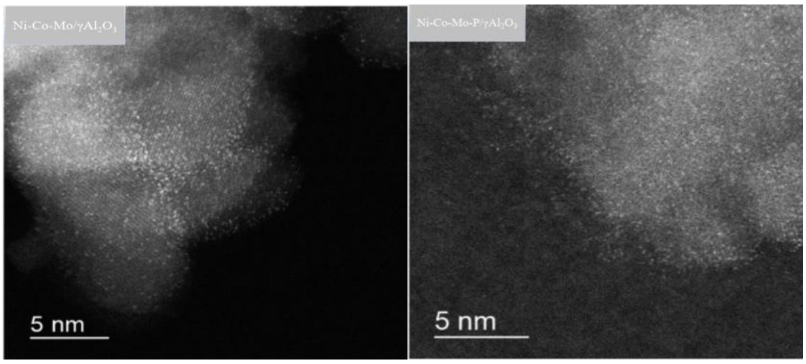


Fig. 7. TEM images of the Ni-Co-Mo/Al_2O_3 and Ni-Co-Mo-P/Al_2O_3 catalysts

As shown in the figure, after loading active components (Ni, Co, Mo, P) onto the Al_2O_3-based carrier, a greater number of fine white particles appeared on the carrier surface. This phenomenon is explained by the successful adsorption of the active particles onto the Al_2O_3 and their integration into the carrier surface. The high-density structure demonstrated the interaction between the active metal and the carrier.

According to the analysis of the TEM images shown in Figures 7 and 8, the fine crystal structure of the multilayered active particles in the catalyst structure is prone to forming an amorphous metal agglomeration in the dark region. It can be concluded that the dark regions, which appear as single- or multi-layered metals and their oxides, are formed when the amount of active particles increases. Moreover, as the number of active particles grows, more dark spots are observed in the TEM images. As the loading of active particles increased, the intensity level rose, the dispersion of Ni, Co, Mo, and their oxides decreased, and their larger agglomeration on the support surface resulted in larger particle sizes and more active metal reduction. This affects the dissociation and fragmentation properties.

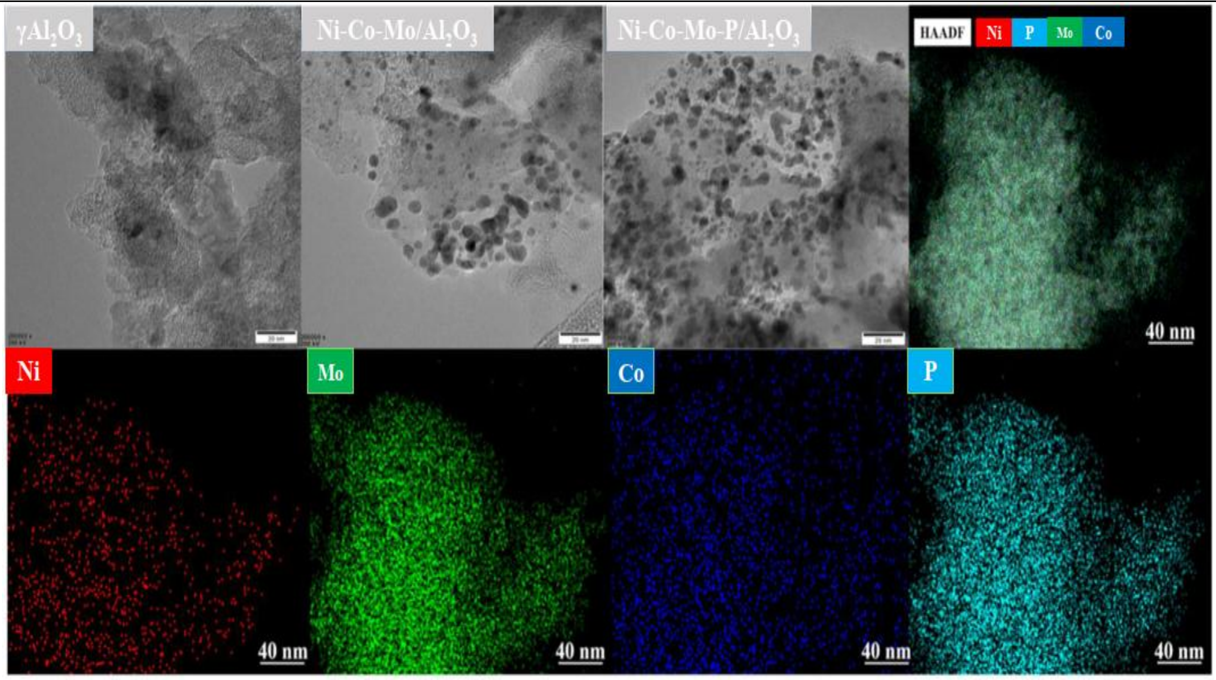


Fig. 8. TEM image of the Ni-Co-Mo-P/Al_2O_3 catalyst

TEM images of all the studied catalyst samples show Ni, Co, Mo, and Ni, Co, Mo, P particles adsorbed on the carrier surface and their typical structure [27–29]. They appear as filament-like layers and multiple layers. When their basal planes are oriented parallel to the electron beam, they are observed as dark lines. As is known, Mo compounds are a layered phase with a hexagonal crystal structure. As is known, molybdenum compounds are layered phases with a hexagonal crystal structure, which is typical for Mo-based catalytic systems [30; 31]. The micrographs showed the presence of Ni, Co, Mo, and their oxide particles in the catalysts. In the micrographs, the black filamentary lines with an interplanar spacing of 0.6 nm correspond to the layers of Ni, Co, and Mo crystallites (Figure 8).

The Ni, Co, and Mo particles in the Ni-Co-Mo-P/Al_2O_3 catalysts appeared as dark spots with clear contrast. Statistical results indicate that the dispersed Ni, Co, and Mo particles on the surface of Al_2O_3 in the reduced Ni-Co-Mo-P/Al_2O_3 catalyst have average particle sizes of 4.3, 5.2, and 5.9 nm, respectively. Active particles and phosphate anions are adsorbed on the oxide phases of the carrier (Al_2O_3). A wider distribution of Co and Mo particles by size and the presence of large agglomerates smaller than 100–500 nm were observed. TEM images are shown to describe the morphological characteristics of the carrier and the distribution of the metals Ni, Co, and Mo in the Ni-Co-Mo-P/Al_2O_3 catalysts. As shown in the figure, the various morphologies of the Al_2O_3 carrier were successfully synthesized by the impregnation method. The dispersion of active particles increased for Ni-Co-Mo/Al_2O_3 (0.25) and Ni-Co-Mo-P/Al_2O_3 (0.26). The dispersion of Ni-Co-Mo in Ni-Co-Mo-P/Al_2O_3 is due to the strong interaction between phosphorus and aluminum. The presence of phosphorus promotes the faster migration of metal precursors into the core.

The textural properties of the Al_2O_3-based catalysts were investigated using low-temperature nitrogen adsorption on an Autosorb-1 (Quantachrome) porosimeter. In this method, the textural properties were investigated by studying the deactivation of the sample and kerogen at controlled pressure using the nitrogen adsorption–desorption technique. The specific surface area of the catalysts was calculated using the Brunauer–Emmett–Teller (BET) model at relative pressure P/Po = 0.05–0.3. Based on the desorption curve, the pore size distribution and their total volume were determined using the Barrett–Joyner–Halenda (BJH) model.

The textural properties of the synthesized catalysts, including pore volume, the distribution of pores on the carrier surface, and specific surface areas, were investigated using N_2 adsorption-desorption isotherms. The N_2 adsorption-desorption isotherms of the synthesized catalysts are presented in Figure 9.

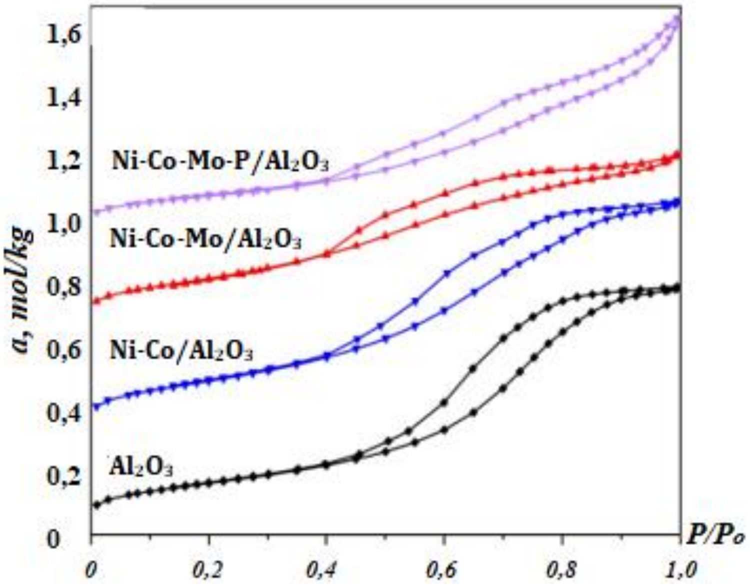


Fig. 9. N_2 adsorption–desorption isotherms of Al_2O_3-based catalysts

Table 4

Porosity distribution of the synthesized catalyst

Catalyst samples	Pore volume (sm³/g)		
	Micropore (d_p < 2 nm)	Mesopore (2 nm < d_p < 50 nm)	Macropore (d_p > 50 nm)
Al_2O_3	-	0.499 (97.0%)	0.016 (3.0%)
Ni-Co/Al_2O_3	-	0.140 (98.4%)	0.002 (1.6%)
Ni-Co-Mo/Al_2O_3	-	0.082 (94.0%)	0.005 (6.0%)
Ni-Co-Mo-P/Al_2O_3	-	0.101 (95.1%)	0.005 (4.9%)

From the isotherm in this figure, it should be noted that, Al_2O_3, The H1-type hysteresis loop was observed for the Ni-Co/Al_2O_3 and Ni-Co-Mo/Al_2O_3 samples. For these three samples, the presence of highly ordered cylindrical mesopores with a narrower pore distribution was identified. Although the hysteresis loop shape in the Ni-Co-Mo-P/Al_2O_3 sample remained Type H1, the parallelism of the adsorption-desorption isotherms decreased. The existence of in-plane mesopores and the shape of the hysteresis loop were observed to be of the H1 type in the low-pressure region and of the H4 type in the high-pressure region. This indicated the presence of two types of mesoporosity: ordered cylindrical mesopores and disordered mesopores between various active particles. It was determined that all samples have a mesoporous structure with cubic and catacomb-like morphological characteristics. As can be seen from the obtained results, the P/Po value of 0.4 in the samples confirms the presence of a mesoporous structure, and the P-Po value of 0.8 proves that the catalysts are mesoporous. Table 4 presents the results of the pore size distribution for the initial carrier Al_2O_3 and the catalysts synthesized on its basis.

Based on the results of the above studies, it can be seen that in all samples of the synthesized catalyst, the diameters of the micro- and mesopores are in the range of 1–2 nm and 2–16 nm, respectively.

Based on the results obtained from determining the N_2 adsorption–desorption isotherms of the synthesized catalysts, the textural properties of these catalysts were determined, and the results are presented in Table 5.

Table 5

Textural properties of the synthesized catalysts

Catalyst samples	Textural properties	
	Surface reference area (S), m²/g	Bulk density of pores (V), sm³/g
γAl_2O_3	302	0.84
Ni-Co/γAl_2O_3	295	0.69
Ni-Co-Mo/γAl_2O_3	279	0.58
Ni-Co-Mo-P/γAl_2O_3	275	0.52

Conclusion

Thus, based on the results of studies on regenerating the used Al_2O_3 adsorbent at high temperatures without the presence of air oxygen, the adsorbent was regenerated at 600 °C for 360 minutes, during which the adsorbent lost the maximum amount of weight, and this condition was adopted as the optimal for the complete regeneration of the used adsorbent.

Based on the analysis of the elemental composition of the regenerated Al_2O_3 adsorbent, it was determined that its composition is 81.5 % Al_2O_3. An adsorbent with this composition was recommended for obtaining bifunctional catalyst samples by impregnating active metal components and was used in the subsequent stages of the study.

Using regenerated Al_2O_3 as a carrier, $(NH_4)_6Mo_7O_{24}·4H_2O$, a process model was developed to synthesize a bifunctional catalyst using salts such as $Ni(NO_3)_2·6H_2O$ and $Co(NO_3)_2·6H_2O$ and H_3PO_4 acid, and based on this model, three types of catalysts were prepared: Ni-Co/Al_2O_3, Ni-Co-Mo/Al_2O_3, and Ni-Co-Mo-P/Al_2O_3 samples were synthesized.

To evaluate the effect of the loading amount of the active components Ni, Co, Mo, and P on the catalyst support, SEM and TEM analyses were conducted. Their results showed that the active metals were uniformly distributed along the radial direction of the microspheres., This demonstrates that this adsorption method can ensure uniform loading for hundreds of micron-sized spheres.

Based on the results of determining the textural properties of the Al_2O_3-based catalysts, their specific surface area is 275-295 m^2/g. The specific pore volume was determined to be 0.52-0.69 cm^3/g. These indicators were proven to be similar to the properties of conventional catalysts.

Based on the results of the conducted studies, the catalysts obtained by impregnating an Al_2O_3-based support with the active components Ni, Co, Mo, and P were recommended for use as bifunctional catalysts in the hydrotreating and hydrogenation processes of petroleum and gas products.

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