

ВЛИЯНИЕ ПОРЯДКА ВВЕДЕНИЯ Со-Мо-СОДЕРЖАЩЕГО АКТИВНОГО КОМПОНЕНТА НА ФИЗИКО-ХИМИЧЕСКИЕ СВОЙСТВА АЛЮМООКСИДНЫХ СИСТЕМ

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В ходе исследований был синтезирован ряд образцов нанесенных металлсодержащих катализаторов методом пропитки с избытком пропиточного раствора с различным порядком введения активных компонентов. Исследование полученных образцов позволило выявить влияние порядка введения активных компонентов на физико-химические и каталитические свойства образца. В качестве источников активных металлов использовались нитрат кобальта и молибденовая синь, синтезированная методом предварительной механоактивации молибденсодержащего прекурсора. Синь также выступала источником структурообразующего металла. Механоактивация проводилась в относительно мягких условиях (ускорение мелющих тел = 5G), что упрощает синтез систем. В роли носителя выступал промышленный порошок псевдобемита, из которого путем температурной обработки получали γ - Al_2O_3 . Полученные системы были исследованы методами ИК-спектроскопии и сканирующей электронной микроскопии. ИК спектры показали схожие профили систем, в обоих случаях термообработка позволила почти полностью элиминировать азотсодержащие компоненты. Удаление азотсодержащих компонентов фиксировали по уменьшению интенсивности полосы поглощения в области 1380-1400 см^{-1} . По результатам сканирующей электронной микроскопии было выявлено, что способ введения активного компонента влияет на текстурные характеристики, значительно меняется площадь удельной поверхности, однако термообработка при 400 °С нивелирует этот эффект. На основании данных обработки около 1000 частиц был высчитан средний размер частиц для каждого образца. Было выявлено, что в зависимости от порядка введения этот параметр различается почти вдвое. Таким образом можно сделать вывод, что порядок введения не оказывает значительного влияния на химический состав поверхности, большим изменениям подвергаются некоторые текстурно-морфологические свойства.

Ключевые слова: оксид алюминия, полиоксометаллаты, молибденовая синь, гидроочистка, гидродесульфирование

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INFLUENCE OF THE ORDER OF INTRODUCTION OF A Co-Mo-CONTAINING ACTIVE COMPONENT ON THE PHYSICOCHEMICAL PROPERTIES OF ALUMINUM OXIDE SYSTEMS

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During the research, a number of samples of supported metal-containing catalysts were synthesized by impregnation with an excess of impregnating solution with different orders of introducing active components. The study of the obtained samples revealed the effect of the order of introducing active components on the physicochemical and catalytic properties of the sample. Cobalt nitrate and molybdenum blue synthesized by preliminary mechanical activation of a molybdenum-containing precursor were used as sources of active metals. The blue also served as a source of structure-forming metal. Mechanical activation was carried out under relatively mild conditions (acceleration of grinding bodies = 5G), which simplifies the synthesis of the systems. Industrial pseudoboehmite powder served as a carrier, from which γ -Al₂O₃ was obtained by heat treatment. The obtained systems were studied by IR spectroscopy and scanning electron microscopy. The IR spectra showed similar profiles of the systems; in both cases, heat treatment allowed almost complete elimination of nitrogen-containing components. The removal of nitrogen-containing components was recorded by a decrease in the intensity of the absorption band in the region of 1380-1400 cm⁻¹. Scanning electron microscopy showed that the method of introducing the active component affects the textural characteristics, the specific surface area changes significantly, but heat treatment at 400 °C neutralizes this effect. Based on the data from processing about 1000 particles, the average particle size for each sample was calculated. It was found that depending on the order of introduction, this parameter differs almost twofold. Thus, it can be concluded that the order of introduction does not significantly affect the chemical composition of the surface, some textural and morphological properties are subject to significant changes.

Keywords: aluminum oxide, polyoxometalates, molybdenum blue, hydrotreating, hydrodesulfurization

INTRODUCTION

The study of various methods for preparing catalytic systems used in petroleum refining is an important aspect in the field of hydrocarbon raw material processing [1-4]. The development of technologies in this area will significantly improve the quality of the products obtained, involve heavier oils and achieve a higher degree of conversion of the initial petroleum components into the final product. Wide distribution, a huge range of applications and a high grade of flexibility continue to fuel the interest of researchers around the world. At the same time, the degree of development of native catalysts for some processes is significantly lower than the global average, so conducting research in this area is an urgent task. An important stage in the creation of a catalyst is synthesis, due to the huge number of factors and conditions, varying which it is possible to achieve the required properties dictated by the

specific process for which this catalyst is created [5]. One of the important stages of synthesis is the order of introduction of the active component onto the surface of the carrier. It is known that depending on the order of introduction, the morphology of the carrier or active phase particles, their size and degree of dispersion can significantly change, which in turn also affects the final catalytic activity [6]. Therefore, to study this issue, a number of systems were synthesized, differing in the order of introduction of active components. Also important is the choice of the active component, which in the case of catalysts for hydroprocesses are compounds of transition metals, most often molybdenum/tungsten sulfides, applied to various types of carriers with different degrees of acidity (aluminum oxides, aluminophosphate composites, carbon carriers), promoted with cobalt and/or nickel [7-9]. Such systems have proven themselves well in industrial applications.

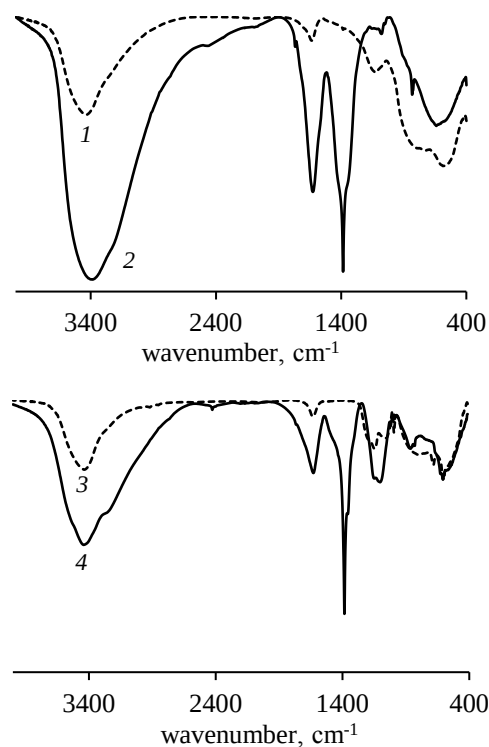


Fig. 1. IR-spectroscopy results (1 – Con 400, 2 – Con 25, 3 – Par 400, 4 – Par 25)

Рис. 1. Результаты ИК-спектроскопии (1 – Con 400, 2 – Con 25, 3 – Par 400, 4 – Par 25)

EXPERIMENTAL PROCEDURE

Aluminum oxide ($\gamma\text{-Al}_2\text{O}_3$) was chosen as a support for the catalytic systems. It was chosen due to its satisfactory textural properties, as well as its widespread occurrence and availability. To obtain the support, industrial pseudoboehmite powder – ALOOH (OOO Ishimbay Specialized Chemical Catalyst Plant) was used, which was calcined in a muffle furnace at $T = 550^\circ\text{C}$ for 4 h [10]. To prepare an impregnation solution for obtaining systems with the simultaneous introduction of metals, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (chemically pure) was used as a Co-containing component in an amount of 3% by weight (in terms of metal), which was dissolved in an alcohol solution of molybdenum blue [11]. For sequential introduction, an alcohol solution of salt was prepared based on the same cobalt nitrate, and impregnated with molybdenum blue at the next stage. After each impregnation, the system was calcined at 400°C for 4 h. Molybdenum blue was synthesized using an original technique using a pre-mechanically activated MoS_2 precursor [12]. Samples obtained at room temperature without heat treatment serve as a comparison point for studying the genesis of active component precursors [13]. Samples with simultaneous introduction were given the codes "Par 25" and "Par 400", for systems with sequential introduction, the

codes "Con 25" and "Con 400" were assigned. The digital part of the code indicates the heat treatment temperature.

Subsequently, the catalytic systems were studied using IR spectroscopy and scanning electron microscopy (SEM). Infrared spectra were recorded on a Nicolet 5700 IR Fourier spectrometer (USA) in the frequency range of $4000\text{--}400\text{ cm}^{-1}$ with a resolution of 4 cm^{-1} . Solid samples for shooting were prepared according to the standard method by pressing tablets of 3 mg of the powder under study with 300 mg of KBr. The specific surface was determined by the BET method by low-temperature nitrogen adsorption on the Sorbtometer M device from KATAKON. Additionally, the specific surface and volume of micropores were determined on this device by the comparative method. The morphology of the samples was studied on a LEO EVO 50 microscope at an accelerating voltage of 15 kV.

РЕЗУЛЬТАТЫ И ИХ ОБСУЖДЕНИЕ

Fig. 1 shows the IR spectra of the initial samples and those calcined at 400°C with simultaneous and sequential introduction of active components. The absorption bands (AB) in the regions of 1399 cm^{-1} and 1600 cm^{-1} characterize the symmetric and asymmetric stretching vibrations of N-O and N=O. From these spectra it can be concluded that heat treatment at 400°C promotes almost complete elimination of nitrogen-containing components [14, 15]. The presence of nitrogen in the composition of catalysts for hydroprocesses is a negative factor [16], therefore its removal is a necessary step in the synthesis of systems. In this case, in the case of a sample with sequential introduction, the intensity of the AB in the region of 1399 cm^{-1} is higher than that of the sample with simultaneous introduction, which may indicate a higher concentration of the impregnating solution that got onto the carrier, however, upon calcination, this band disappears, while the absorption bands characterizing the carrier remain in the spectrum: absorption bands $515\text{--}560\text{ cm}^{-1}$ and a wide band in the region of $850\text{--}900\text{ cm}^{-1}$ relate to the stretching vibrations of Al-O and Al-O-Al [17]. The band in the region of 3400 cm^{-1} , common to all systems, relates to the stretching vibrations of the OH group of adsorbed water molecules [18]. It is also possible to assume the presence of absorption bands Mo-O and O-Mo-O, characterizing the deposition of molybdenum oxide structures in the region of low wave numbers $900\text{--}1100\text{ cm}^{-1}$ [19], however, their extremely low intensity (due to the low concentration) does not allow them to be confidently identified and requires additional research. The absorption bands in the region of $950\text{--}1200\text{ cm}^{-1}$ may also be related to cobalt oxide structures formed as a result of thermal decomposition

of the initial nitrate [20]. The order of introduction also affects the textural characteristics. For comparison, the parameters of the initial gamma oxide were also measured. The results are presented in Table.

The data in the table shows that the order of introduction of the systems that have not undergone heat treatment, the specific surface area (S_{sp}) differs significantly ($\Delta = 32,4 \text{ m}^2/\text{g}$). At the same time, after heat treatment, S_{sp} is almost the same for both systems, and is less than that of the original carrier, which indicates the application of active components to the carrier. The fact of successful application is also confirmed by the results of scanning electron microscopy.

Table
Textural properties of samples (S_{sp} - the specific surface area, V_{total} - total volume of pores, D - pore diameter)

Таблица. Текстуальные свойства образцов
($S_{уд}$ - удельная площадь поверхности, $V_{общ}$ - общий объем пор, D - диаметр пор)

Sample	S_{sp} , (m^2/g)	V_{total} , (cm^3/g)	D , nm
Al_2O_3	189,9	0,086	1,819
Con-25	148,7	0,070	1,886
Par-25	116,3	0,052	1,832
Con-400	160,8	0,075	1,876
Par-400	159,7	0,074	1,870

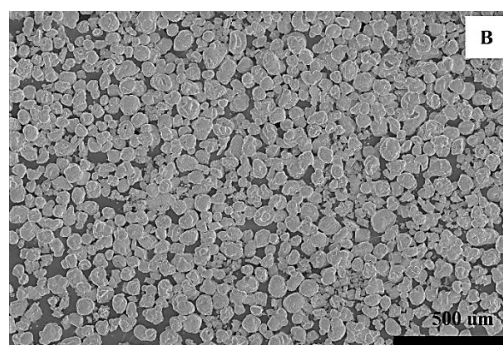
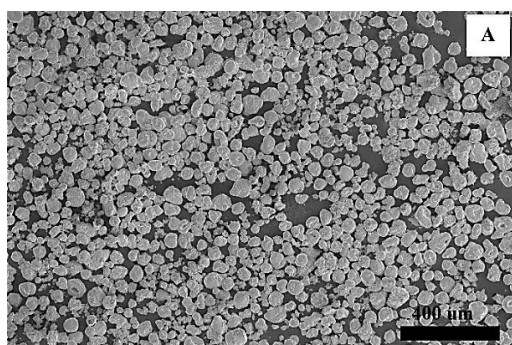


Fig. 2. Microphotographs of samples (A - Par 25, B- Par 400)
Рис. 2. Микрофотографии образцов (A - Par 25, B - Par 400)

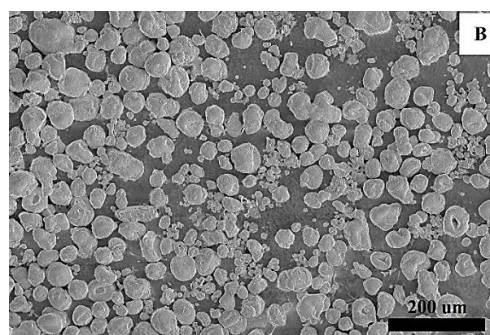
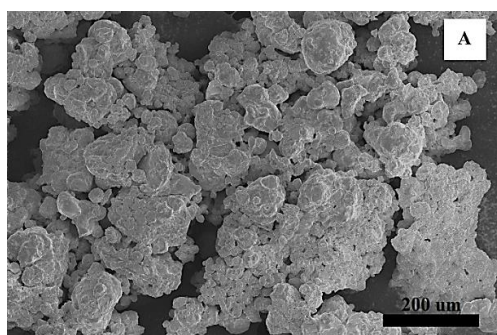


Fig. 3. Microphotographs of samples (A - Con 25, B- Con 400)
Рис. 3. Микрофотографии образцов (A - Con 25, B - Con 400)

Fig. 2 and 3 show the scanning electron microscopy results. Morphologically, the particles both before and after heat treatment are slightly differs in case of parallel type of active component adding. In the second case, large differences are observed. Thus, before calcination, the particles form large agglomerates. However, calcination leads to their destruction, after which the gap in morphology disappears. Shape of particles is a sphere-like with a defective surface, some of which contain cavities of various sizes. After processing the SEM images, granulometric analysis of the particles was carried out. To generate data on the distribution of fractions by size > 1000 particles were processed. The order of introduction affected the frac-

tional composition; in the case of simultaneous introduction, regardless of heat treatment, the fractional composition is represented mainly by particles with a size of 0-100 μm , with a predominance of the 50-100 μm fraction. The average particle size is 55 μm . In the case of sequential introduction of active metals, the fractional distribution looks somewhat different, the predominant fraction is particles of 0-50 μm , and the proportion of the 50-100 μm fraction decreases. The average particle size also decreases naturally - 44.94 μm before heat treatment and 38.57 μm after. Such differences may be caused by an increase in the number of processing stages at the synthesis [21].

CONCLUSION

Thus Co-Mo/ γ -Al₂O₃ catalytic systems were synthesized and studied. It was shown that molybdenum blues can be used as a Mo-containing reagent, while the sequential introduction of active metals leads to an insignificant change in the shape of the particles, while the resulting particles have a smaller average size, and the predominant fraction is particles of 0-50 μ m. The specific surface area of the resulting systems also significantly depends on the order of introduction, but heat treatment levels out these differences. At the same time, according to IR spectroscopy, the chemical composition of the surface does not undergo significant changes.

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