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КАТАЛИЗ ЖӘНЕ ЭЛЕКТРОХИМИЯ ИНСТИТУТЫ» АҚ

# Х А Б А Р Л А Р Ы

## ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК  
РЕСПУБЛИКИ КАЗАХСТАН

АО «ИНСТИТУТ ТОПЛИВА, КАТАЛИЗА И  
ЭЛЕКТРОХИМИИ ИМ. Д.В. СОКОЛЬСКОГО»

## NEWS

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OF THE REPUBLIC OF KAZAKHSTAN

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## ХИМИЯ ЖӘНЕ ТЕХНОЛОГИЯ СЕРИЯСЫ

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НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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**INTERACTION OF THE Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> CATALYTIC SYSTEM  
WITH PROBE MOLECULES I. RESEARCH OF THE  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>  
AND THE Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> INITIAL SYSTEM**

**Abstract.** The work is the first part of the studies devoted to the interaction of a heterogeneous catalytic system with adsorbed molecules. It presents the results for the initial oxide  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> and Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system with an iron content of 0.5; 3; 13% by weight, obtained with a wide range of physicochemical methods. The Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system was chosen as the object of the study, since it exhibits catalytic activity in many chemical processes and can later be used as a model.

The performed work showed that during the preparation of the Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system by impregnation, the structure of the support can be modified. The nature of the filling of the support surface with the iron-containing phase depends substantially on its percentage and can be multilayered.

It is established that the Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system contains iron in the form of Fe<sup>3+</sup>. Depending on the iron content, iron-containing aggregates of various sizes may be present in it, both in the paramagnetic and in the magnetically ordered states.

**Key words:** heterogeneous catalysis, physicochemical methods of investigation.

### **Introduction**

This work is the beginning of a series of studies devoted to the interaction of a heterogeneous catalytic system with adsorbed molecules. This formulation of the problem is topical, since, in heterogeneous catalysis, the most important stage is the adsorption stage [1, 2].

The iron-containing Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> ( $\gamma$ -Al<sub>2</sub>O<sub>3</sub>-support) system was chosen as the object of research because it exhibits catalytic activity in many chemical processes, such as the production of ammonia [3-5] and carbon nanotubes [6, 7], in CO hydrogenation reactions (Fischer-Tropsch synthesis) [8, 9], in the oxidation of hydrogen sulphide to elemental sulfur [1], deep processing of solid fossil and renewable organic raw materials [10], in gasoline reforming reactions [11], and many other reactions [12]. Thus, the catalytic system Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> being practically multifunctional, in the future, can be used as a model system.

### **Experimental**

The Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system with an iron content of 0.5; 3; 13 wt.% was prepared by impregnation [1, 13, 14] of the initial  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> oxide with an aqueous solution of iron acetate, followed by drying and calcination in air.

A wide range of physicochemical methods during the research was used.

*X-ray diffractometry.* The X-ray diffractometer Dron-4-07 with cobalt anode tube was used.

Mode:

- speed 2 deg/min;
- working parameters of the tube: 30 kV, 20 mA.

*Transmission electron microscopy.* Equipment - EM-125K, accelerating voltage of 75 kV.

*Scanning electron microscopy.* JSM 6610 LV, JEOL, Japan is a scanning low-vacuum electron microscope. Accelerating voltage 20 kV.

*BET method* (low-temperature nitrogen adsorption). Equipment - AccuSorb, Micromeritics, USA. The standard procedure. Calcination of the sample at 230-250° C for 3 hours with evacuation. The relative error in determining the specific surface area is  $\pm 5\%$ .

*Mossbauer spectroscopy.* Equipment - MS 1104Em, Russia. The source was cobalt 57 in a chromium matrix with an activity of 100  $\mu$ Ci. The spectra were processed on a PC by the "least squares" method. The isomeric shifts are given in terms of  $\alpha$ -Fe. The temperature of the spectra is 23°C. The shooting mode is "on the skylight". The error in determining the isomer shift (IS) is  $\Delta IS = \pm 0.03$  mm/s; quadrupole splitting (QS)  $\Delta QS = \pm 0.03$  mm/s; relative content (S)  $\Delta S = \pm 2\%$ .

### **Results and Discussion**

The study of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> oxide and Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system was carried out.

#### *X-ray diffractometry.*

X-ray diffraction patterns (Fig. 1) of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> oxide and Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system with various iron contents were obtained.

Following results were obtained:

- Initial  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>:

Reflexes at 4.5641; 2.7959; 2.3981; 2.2883; 1.9767; 1.5258; 1.3950 Å -  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> phase (ASTM 10-424);

- 0.5% Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system:

Reflexes at 4.5641; 2.8032; 2.3815; 2.2891; 1.9781; 1.5210; 1.3994 Å -  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> phase (ASTM 10-424);

Reflexes at 4.8524; 4.3735; 4.3158; 3.3285; 2.4580; 2.3815; 1.8017; 1.7475 Å - gibbsite phase of Al(OH)<sub>3</sub> (ASTM 33-18);

Reflexes at 4.1784; 2.6891; 2.4580 Å -  $\alpha$ -FeO(OH) (goethite) phase (ASTM 29 - 713);

- 3% Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system:

Reflexes at 4.5562; 2.8003; 2.3898; 2.2807; 1.9829; 1.5214; 1.3976 Å - phase  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> (ASTM 10-424);

Reflexes at 4.8579; 4.3698; 4.3230; 3.3264; 2.4580; 2.3898; 1.7978; 1.7501 Å - gibbsite phase of Al(OH)<sub>3</sub> (ASTM 33-18);

Reflexes at 4.1784; 2.6904; 2.4580 Å -  $\alpha$ -FeO(OH) (goethite) phase (ASTM 29 - 713)

- 13% Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system:

Reflexes at 4.5681; 2.7974; 2.3908; 2.2853; 1.9733; 1.5217; 1.3994 Å -  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> phase (ASTM 10-424);

Reflexes at 4.8434; 4.3771; 4.3158; 3.3161; 2.4481; 2.3908 Å - gibbsite phase of Al(OH)<sub>3</sub> (ASTM 33-18);

Reflexes at 4.1817; 2.6837; 2.4481 Å -  $\alpha$ -FeO(OH) (goethite) phase (ASTM 29-713).

The data obtained indicate that the original alumina is indeed  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>. Partial hydrolysis of aluminum oxide occurs during the synthesis of the Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system, resulting in the formation of aluminum hydroxide in the form of gibbsite. It should be noted that the highest degree of hydrolysis is observed for the Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system with the lowest iron content and it decreases monotonically with its growth (Fig. 1). This is probably due to the fact that as the concentration of iron acetate increases, in the solution there is an ever-decreasing amount of free water capable of causing hydrolysis of aluminum oxide [15].

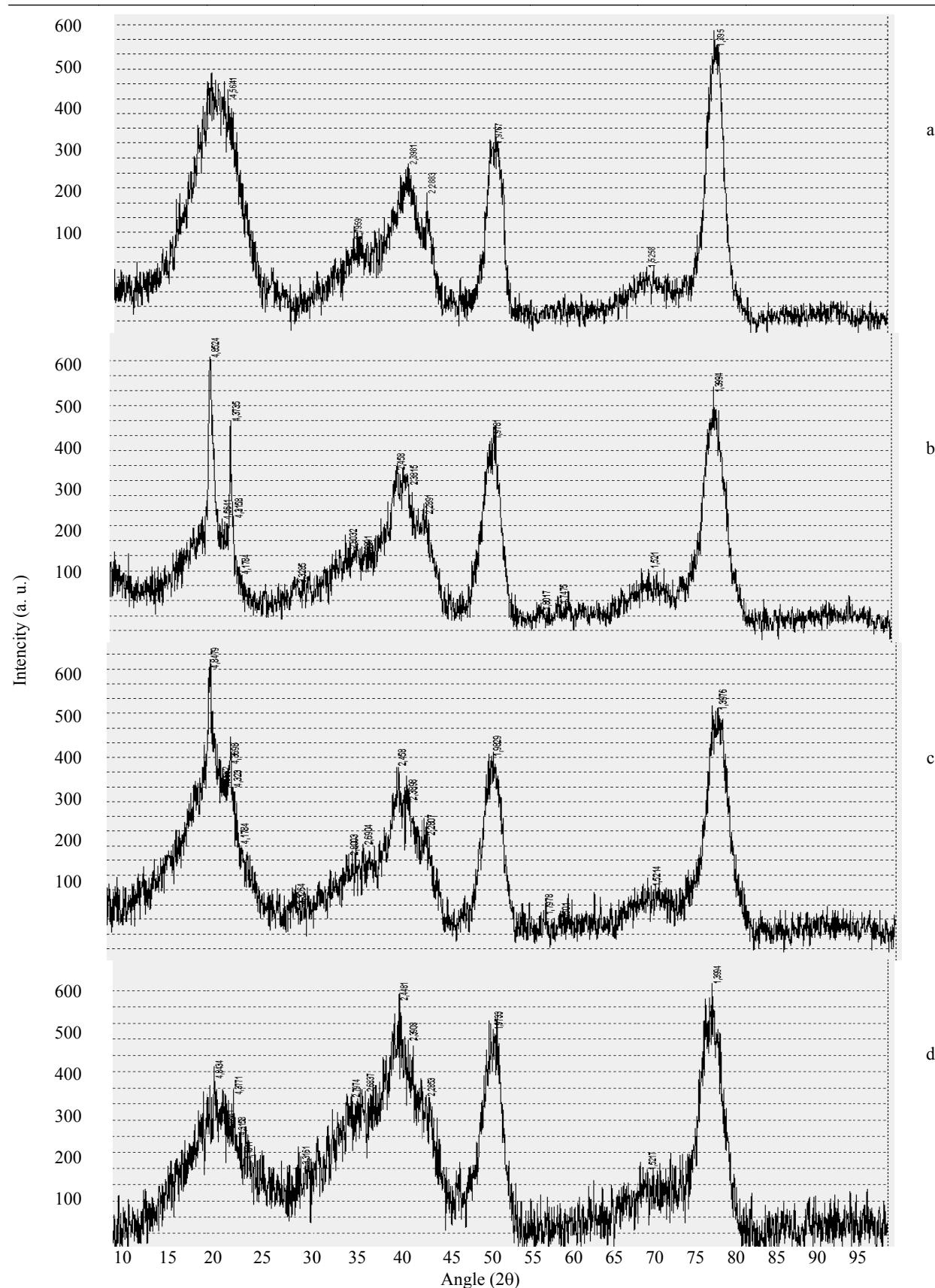


Figure 1 – Diffraction patterns of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> oxide and Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system  
 a -  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>; b - 0.5% Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>; c - 3% Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>; d - 13% Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>

*Transmission electron microscopy.*

Microphotographs of the initial aluminum oxide  $\gamma\text{-Al}_2\text{O}_3$  and the Fe/ $\gamma\text{-Al}_2\text{O}_3$  system with different iron contents are shown in Fig. 2.

There are two kinds of particles in  $\gamma\text{-Al}_2\text{O}_3$  sample. The first type is characterized by aggregates of large, semitransparent, plate-like particles with a hexagonal facets (the (111) plane of  $\alpha\text{-Al}_2\text{O}_3$  crystallites) reaching almost micron sizes (400-800 nm) (Fig. 2a).

The second type of particles is small transparent plates of elongated rectangular shape (planes (001), (111) of  $\gamma\text{-Al}_2\text{O}_3$  crystallites) which in the width are 5-10nm (Fig. 2b). Microdiffraction was taken from various parts of the sample. The pictures show reflexes that are located on the rings. They correspond to  $\gamma\text{-Al}_2\text{O}_3$  (JCPDS, 10-425). A trace amount of  $\delta\text{-Al}_2\text{O}_3$  (JCPDS, 16-394),  $\text{Al(OH)}_3$  gibbsite (JCPDS, 7-324) and  $\theta\text{-Al}_2\text{O}_3$  (JCPDS, 11-517) also were observed.

Micrograph of the 0.5%Fe/ $\gamma\text{-Al}_2\text{O}_3$  system is shown in Fig. 2c. It has large particles with signs of faceting ranging in size from 200 to 1000 nm. Microdiffraction produces symmetrical and individual reflexes, which correspond to  $\gamma\text{-Al}_2\text{O}_3$  (JCPDS, 10-425). A large 200nm in size particle of a pointed shape and small aggregates made up of particles of 10-40 nm in size are shown in Fig. 2d. Microdiffraction gives a large number of symmetrical and individual reflexes, which can be attributed to a mixture of phases: gibbsite (JCPDS, 7-324),  $\beta\text{-AlO(OH)}$  Diaspore (JCPDS, 5-355),  $\gamma\text{-AlOOH}$  Boehmite (JCPDS, 21-1301 ), with a significant predominance of the former.

Only phases of aluminum oxides and hydroxides in the sample were found. The low iron content in the system did not allow the detection of iron-containing phases because of a small amount they did not reach the field of view of the microscope.

The microphotographs of the 3% Fe/ $\gamma\text{-Al}_2\text{O}_3$  system have many dense aggregates; also large particles of lamellar type of medium transparency are present in the sample, which is shown in Fig. 2e. A conglomerate of transparent particles of lamellar type of micron size, surrounded by small aggregates of small dense particles 5-10 nm in size is shown in Fig. 2e. Microdiffraction gives a large set of reflexes that are located along a distorted hexagonal view, and individual reflexes, which can be attributed to a mixture of the phases  $\gamma\text{-FeO(OH)}$  - goethite (JCPDS, 29-713),  $\text{FeOOH}$  (JCPDS, 26-792),  $\text{Al(OH)}_3$  - gibbsite (JCPDS, 29-41),  $\gamma\text{-Al}_2\text{O}_3$  (JCPDS, 10-425).

There is an aggregate of transparent particles of lamellar type ~ 0.5 micron in size for a 13%Fe/ $\gamma\text{-Al}_2\text{O}_3$  system in Fig. 2g. Microdiffraction gives reflexes located on a distorted hexagonal view, which can be referred to  $\delta\text{-FeOOH}$  (JCPDS, 13-87). In addition, this micrograph has round-shaped particles with dimensions of 10-30nm. Microdiffraction gives reflexes (rings) related to  $\text{FeOOH}$  (JCPDS, 26-792). In Fig. 2h, the particles are larger, for them microdiffraction shows a large set of reflections of the following phases:  $\text{Fe}_2\text{O}_3$  (JCPDS, 32-469) and  $\gamma\text{-Al}_2\text{O}_3$  (JCPDS, 10-425).

*Scanning electron microscopy*

Microphotographs of the initial aluminum oxide  $\gamma\text{-Al}_2\text{O}_3$  and the Fe/ $\gamma\text{-Al}_2\text{O}_3$  system with different iron content are shown in Fig. 3.

From microphotographs it follows that aggregates of various sizes (Fig. 3a, b, c) are present on the surface of the support  $\gamma\text{-Al}_2\text{O}_3$ , from tens to micrometers. Porous structure of the support surface is clearly visible when the magnification is large (Fig. 3c).

The relief of the support is "smoothed" when the acetate of iron is deposited on  $\gamma\text{-Al}_2\text{O}_3$  (Figure 3), and the effect is most pronounced at 13% iron content. It can be assumed that an iron-containing film (crust) is formed on the surface of the support, and this, in fact, leads to a visible "smoothing" effect of the relief. In the case of the Fe/ $\gamma\text{-Al}_2\text{O}_3$  system with 0.5% iron content (Fig. 3d, e, f), the complete surface coverage of the  $\gamma\text{-Al}_2\text{O}_3$  support by the iron-containing component is not noticeable, apparently due to its small amount. On the contrary, it can be assumed that for a system with 13% iron content (Fig. 3 j, k, l), the coating is multilayer.

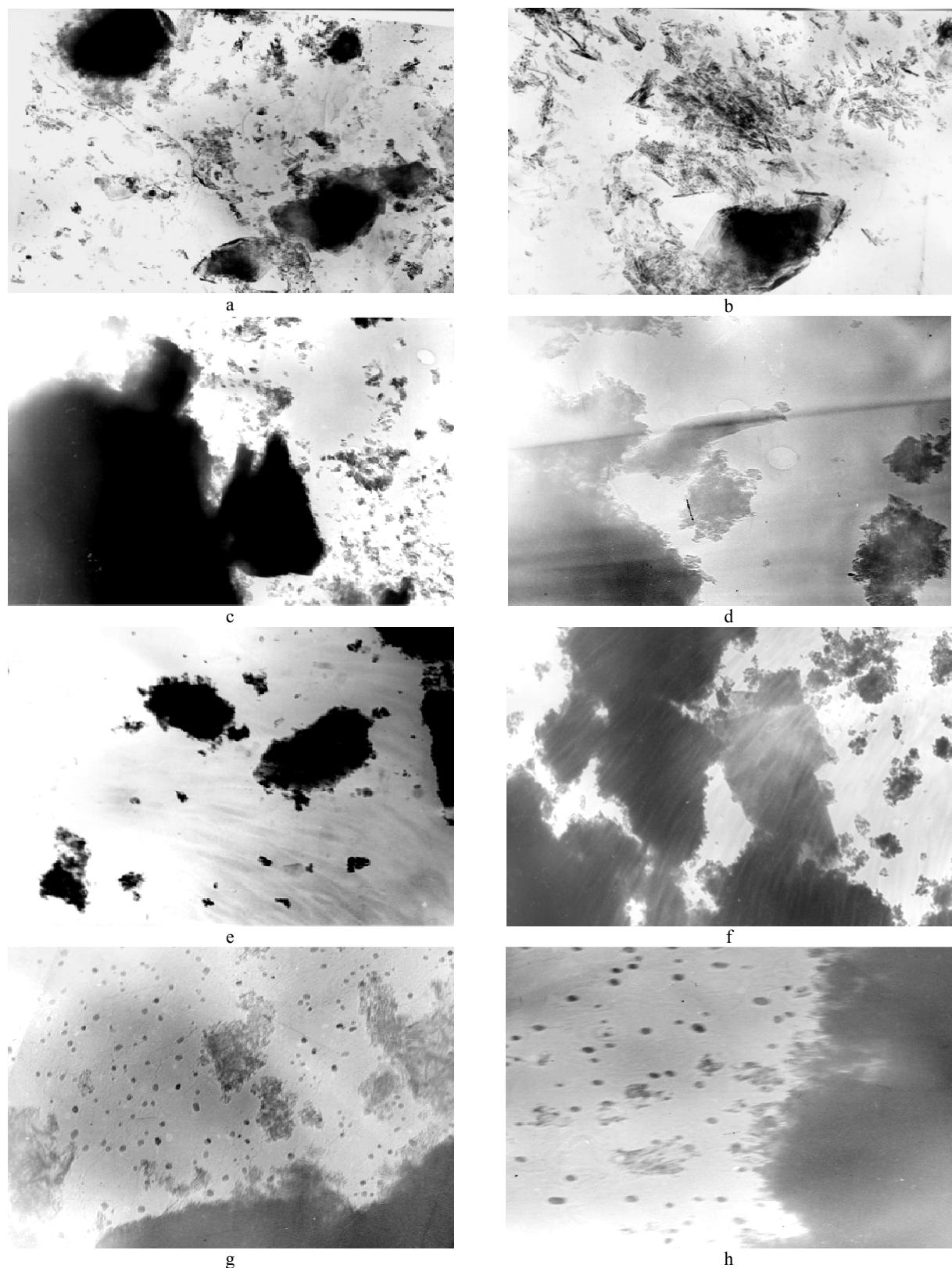


Figure 2 - Micrographs of the initial aluminum oxide  $\gamma$ - $\text{Al}_2\text{O}_3$  and the Fe/ $\gamma$ - $\text{Al}_2\text{O}_3$  system with an iron content of 0.5; 3 and 13%  
a, b - initial  $\gamma$ - $\text{Al}_2\text{O}_3$  oxide; c, d –  $0.5\%$  Fe/ $\gamma$ - $\text{Al}_2\text{O}_3$  system; e, f -  $3\%$  Fe/ $\gamma$ - $\text{Al}_2\text{O}_3$  system; g, h  $13\%$  Fe/ $\gamma$ - $\text{Al}_2\text{O}_3$  system.  
Magnification: a, c, e, f – 24000 times; b, d, g, h – 50000 times

*BET method*

Specific surface area and its texture (porosity) for  $\gamma\text{-Al}_2\text{O}_3$  oxide and Fe/ $\gamma\text{-Al}_2\text{O}_3$  system with different iron content are determined. The results are shown in Table 1 and are shown in Fig. 4. The data for  $\gamma\text{-Al}_2\text{O}_3$  in agreement with the results of [16].

Table 1 – The specific surface area of the  $\gamma\text{-Al}_2\text{O}_3$  oxide and the Fe/ $\gamma\text{-Al}_2\text{O}_3$  system

Sample	Parameter		
	SW, $\text{m}^2/\text{g}$	$V_{\text{ADSmax}}$ , $\text{mL/g}$	$V_{\text{true}}$ , $\text{mL/g}$
$\gamma\text{-Al}_2\text{O}_3$	214	180	0,28
0.5%Fe/ $\gamma\text{-Al}_2\text{O}_3$	211	196	0,31
3%Fe/ $\gamma\text{-Al}_2\text{O}_3$	190	115	0,18
13%Fe/ $\gamma\text{-Al}_2\text{O}_3$	173	101	0,16

Note: SW - specific surface area,  $\text{m}^2/\text{g}$ ;  
 $V_{\text{ADSmax}}$  - total pore volume with gas filling,  $\text{mL/g}$ ;  
 $V_{\text{true}}$  - total true pore volume,  $\text{mL/g}$

From Table 1 it follows that when the iron is precipitated to the support, as the content thereof increases, the value of the specific surface tends to decrease, indicating the filling of the surface.

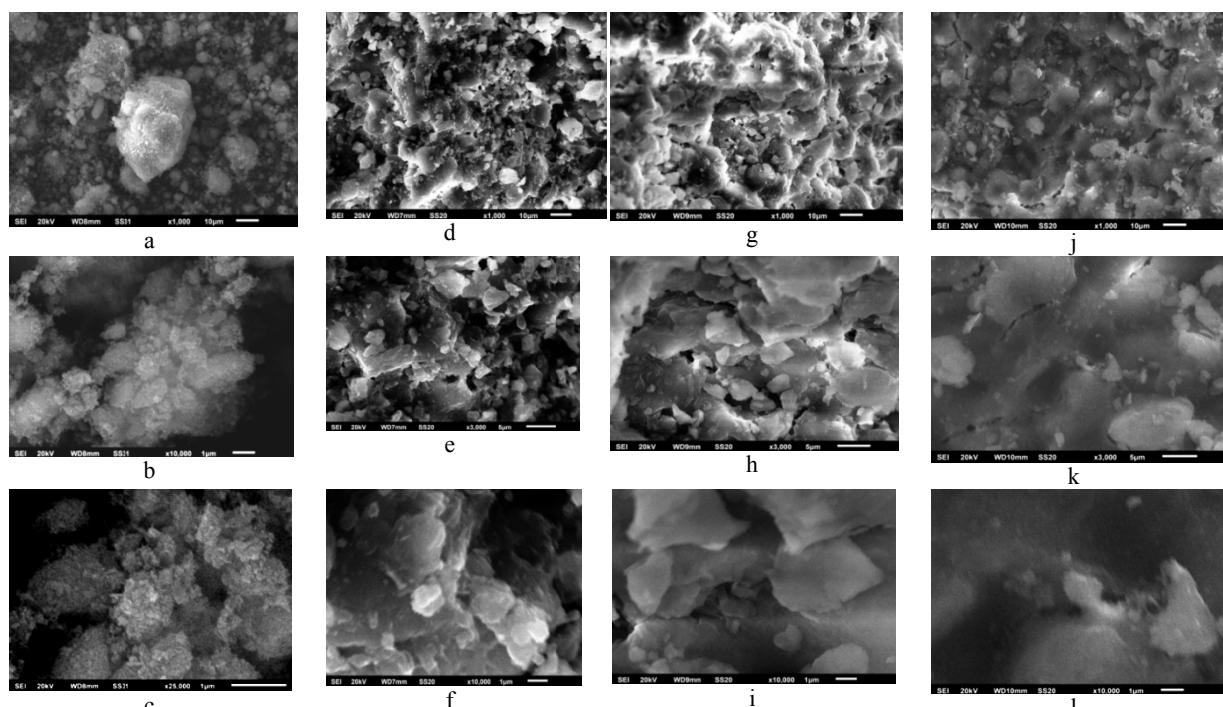


Figure 3 - Microphotographs of aluminum  $\gamma\text{-Al}_2\text{O}_3$  and Fe/ $\gamma\text{-Al}_2\text{O}_3$  systems with different iron content

$\gamma\text{-Al}_2\text{O}_3$  - (a, b, c); 0.5%Fe/ $\gamma\text{-Al}_2\text{O}_3$  - (d, e, f); 3%Fe/ $\gamma\text{-Al}_2\text{O}_3$  - (g, h, i); 13%Fe/ $\gamma\text{-Al}_2\text{O}_3$  - (j, k, l).

Magnification: (a, d, g, j) – 1000 times; (e, h, k) – 3000 times; (b, f, i, l) – 10000 times; (c) – 25000 times

At the same time, the pore volume has a maximum at the point for the system of 0.5%Fe/ $\gamma\text{-Al}_2\text{O}_3$ . The presence of an extremum can probably be explained by the hydrolysis of  $\gamma\text{-Al}_2\text{O}_3$  and the transition of a part of aluminum oxide to the hydroxide, which was discussed above. In aluminum hydroxide, the total pore volume is noticeably higher than that of  $\gamma\text{-Al}_2\text{O}_3$  [17]. Since the iron-containing component does not completely cover the support surface in the 0.5%Fe/ $\gamma\text{-Al}_2\text{O}_3$  system (Fig. 3d, e, f), therefore, at least some of the pores of the hydroxide are accessible.

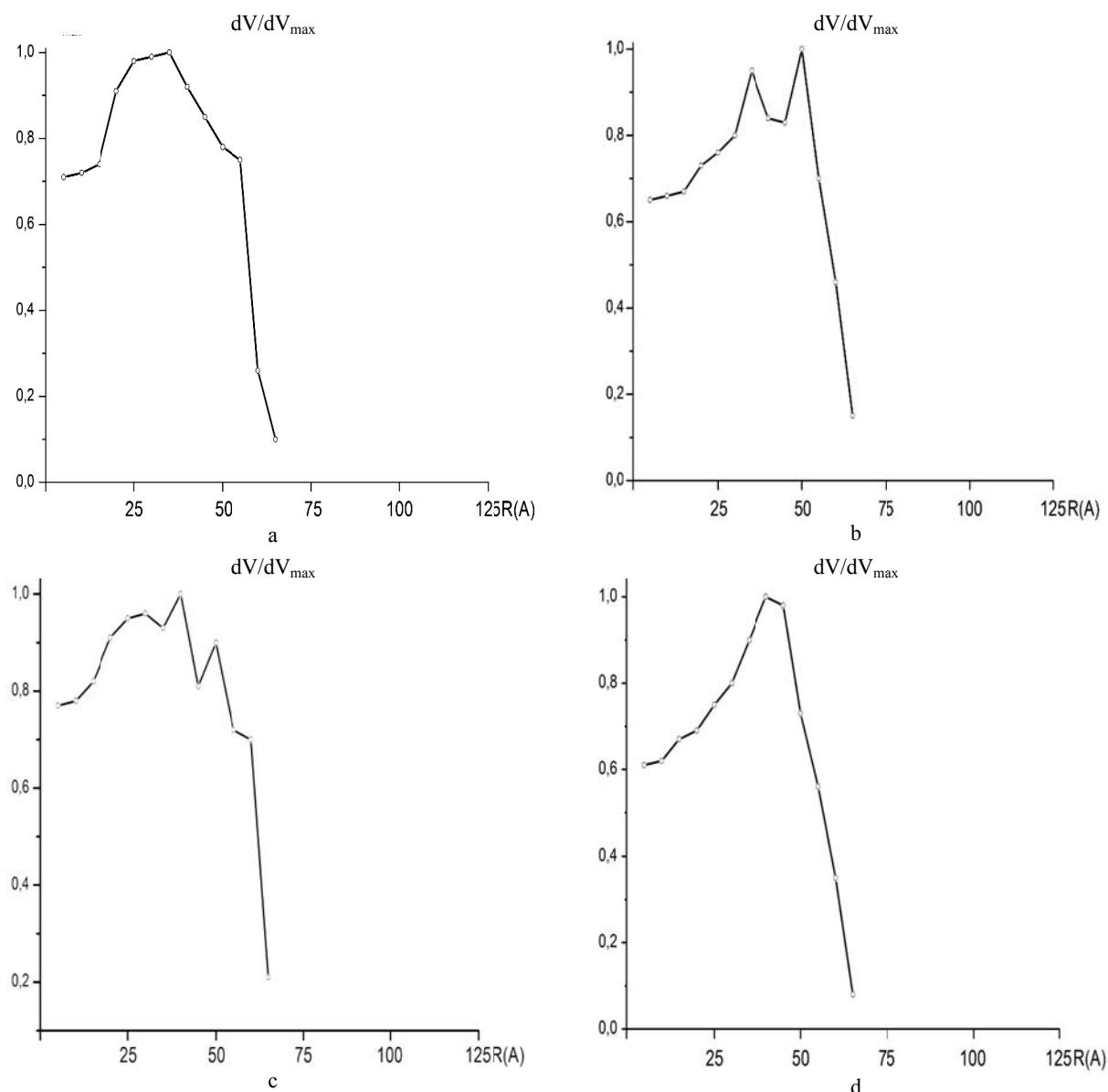


Figure 4 - Pore size distribution in  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> and in the Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system with different iron content  
a -  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>; b - 0.5%Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>; c - 3% Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>; d - 13%Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>

R(Å) is the pore radius in angstroms (Å); dV/dV<sub>max</sub> the ratio  
of the pore volume of a given radius to the maximum volume

The combination of these factors may lead to an increase in the total pore volume for the 0.5%Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system.

With an increase in the iron content the total pore volume decreases which on one side may indicate their possible filling, and on the other hand, the difficulty of accessing the probe gas (nitrogen). The latter can be indicated by scanning electron microscopy data (Figure 3), from which it follows that with a high content of iron in the system, the support surface is almost completely closed.

In Fig. 4 - pore size distribution for  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> and Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>. The pore size for  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> and the Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system fits within a narrow range of values and their radius does not exceed 65-70 Å (Fig. 4). When the iron-containing component is applied to alumina, the pore size distribution in comparison with  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> (Fig. 4a) varies, for 0.5%Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, a "dip" is observed in the 40-45 Å region (Fig. 4b), it follows that in the first place pores of this size are filled. For the 3%Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system, this "gap" is retained, although the shape of the distribution varies (Fig. 4c), which is apparently related to the filling of the surface with

an iron-containing component. For a system of 13%Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, due to the filling of the surface, the access of the probe gas to the pores of small size is essentially limited and the pore size distribution shows the presence of basically larger pores (Fig. 4d). All this occurs against the background of a decrease in the total volume of pores (Table 1).

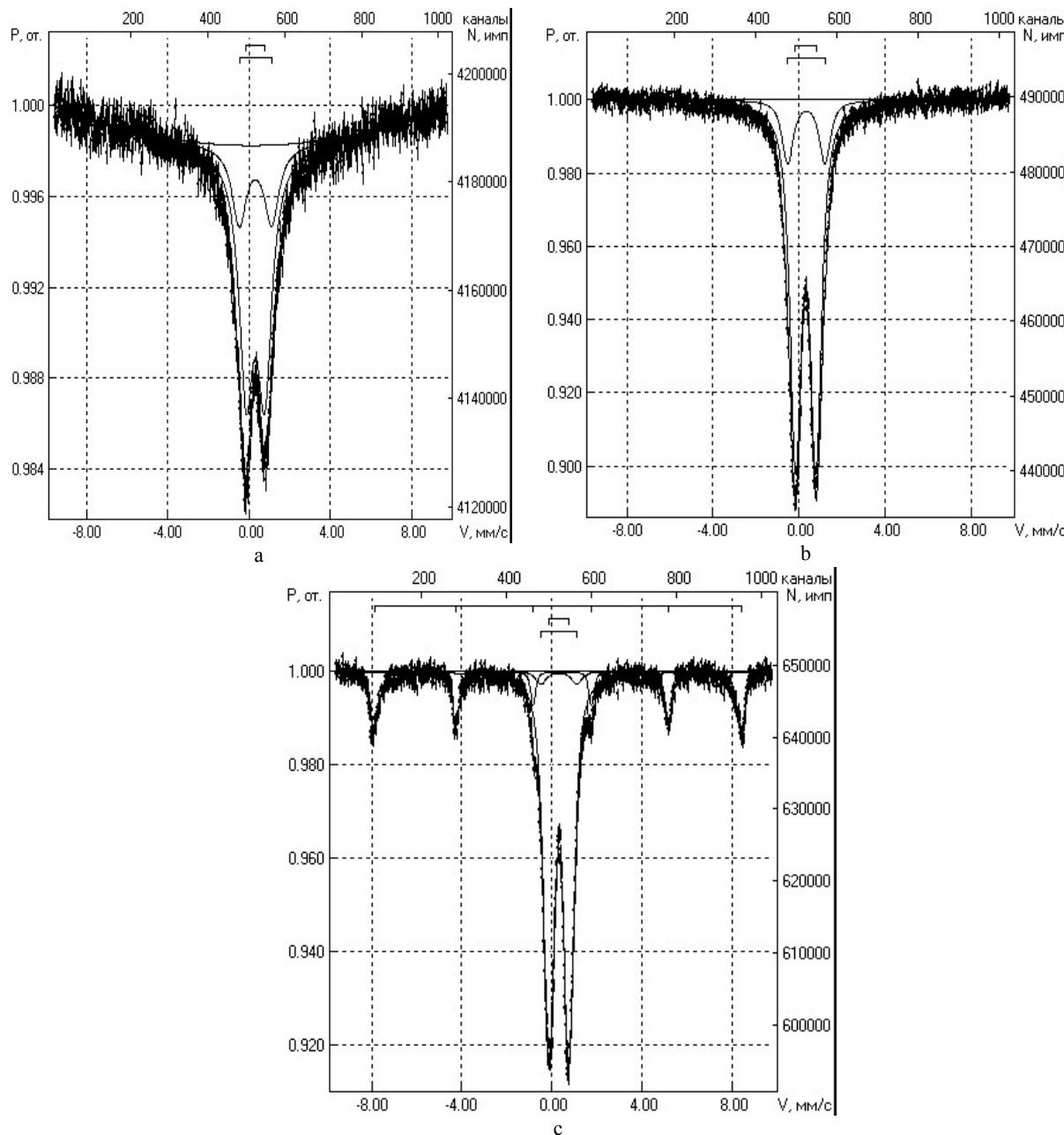


Figure 5 - Mossbauer spectra of the Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system with different iron content  
a – 0,5%Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>; b – 3%Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>; c – 13%Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>

#### *Mossbauer spectroscopy*

Mössbauer spectra of the Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system with different iron contents are shown in Figure 5. The spectra showed that the system, based on the values of isomeric shifts, contains various forms of Fe<sup>3+</sup> both in the paramagnetic state and in the magnetically ordered one.

The following iron forms are present in the system:

0.5%Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>  
Fe<sub>1</sub><sup>3+</sup> form - IS = 0.32 mm•s<sup>-1</sup>; QS = 0.95 mm•s<sup>-1</sup>; S = 62%

$\text{Fe}_2^{3+}$  form - IS = 0.32 mm•s<sup>-1</sup>; QS = 1.59 mm•s<sup>-1</sup>; S = 38%

3%Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>

$\text{Fe}_1^{3+}$  form - IS = 0.31 mm•s<sup>-1</sup>; QS = 0.98 mm•s<sup>-1</sup>; S = 70%

$\text{Fe}_2^{3+}$  form - IS = 0.32 mm•s<sup>-1</sup>; QS = 1.62 mm•s<sup>-1</sup>; S = 30%

13%Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>

$\text{Fe}_1^{3+}$  form - IS = 0.32 mm•s<sup>-1</sup>; QS = 0.93 mm•s<sup>-1</sup>; S = 77%

$\text{Fe}_2^{3+}$  form - IS = 0.31 mm•s<sup>-1</sup>; QS = 1.59 mm•s<sup>-1</sup>; S = 3%

$\alpha$ -Fe<sub>2</sub>O<sub>3</sub> - IS = 0.37 mm•s<sup>-1</sup>; QS = - 0.21 mm•s<sup>-1</sup>; H<sub>eff</sub> = 506 kOe; S = 20%

(H<sub>eff</sub> is the Zeeman hyperfine magnetic splitting)

The forms of  $\text{Fe}_1^{3+}$  and  $\text{Fe}_2^{3+}$ , irrespective of the iron content in the Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system, have similar parameters and are paramagnetic. At the same time, having almost identical IS, they differ significantly in QS. The shape of  $\text{Fe}_2^{3+}$  with a large value of QS can be attributed to iron on the surface of the support, and the form of  $\text{Fe}_1^{3+}$  with smaller QS - to a more deeply located [18, 19].

It should be noted that with an increase in the iron content in the system, a gradual increase in the relative content of the more deeply located form of  $\text{Fe}_1^{3+}$  takes place, which agrees with the BET data.

There is also a magnetically ordered phase that corresponds to  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> in the 13% Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system, in addition to the  $\text{Fe}_1^{3+}$  and  $\text{Fe}_2^{3+}$  forms [20]. A somewhat smaller value of H<sub>eff</sub> (should be 515-517 kOe) indicates that the particles giving in the Mössbauer spectrum of the Zeeman hyperfine magnetic splitting are of the order of 8-12 nm [21]. At such particle sizes, H<sub>eff</sub> has a smaller value than for bulk samples.

## Conclusion

The performed work has shown that during the preparation of the Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system by impregnation, the structure of the support can be modified. The nature of the filling of the support surface with the iron-containing phase depends substantially on its percentage and can be multilayered.

It has been established that the Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> system contains iron in the form of Fe<sup>3+</sup>. Depending on the iron content, iron-containing aggregates of various sizes can be present in the system, both in the paramagnetic and magnetically ordered states.

## Acknowledgments

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**МОЛЕКУЛА ЗОНДЫ БАР Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> КАТАЛИЗДІК ЖҮЙЕНІЦ ӨЗАРА ӘРЕКЕТТЕСТИГІ  
I.  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> ЖӘНЕ Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> БАСТАПҚЫ ЖҮЙЕНІЦ ЗЕРТЕЛЕУІ**

Бұл жұмыс адсорбцияланған молекулаға ие гетерогенді катализдік жүйенің өзара әрекеттестігіне арналған зерттеудің бірінші болігі болып табылады. Оған бастапқы  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> тотығы және 0,5; 3; 13 вес.% темір мөлшері бар Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> жүйесі бойынша кең ауқымды физика-химиялық әдістер жиынтығы қомегімен алынған нәтижелер ұсынылған. Қоңырауда химиялық процестерде катализдік белсенділік көрсетіп, келешекте модельдік ретінде қолдану мүмкіндігі болғандықтан Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> жүйесі зерттеу нысаны болып таңдалған болатын.

Жүргізілген жұмыс сініру әдісімен Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> жүйесін дайындау процесінде тасығыш құрылымының модификациясы мүмкін екендігін көрсетті. Теміркүрамды fazamен тасығыш бетінің толтырылу сипаты айтартылған оның пайыздық мөлшеріне тәуелді және көп қабатты болуы мүмкін.

Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> жүйесі Fe<sup>3+</sup> формалы темір күрайтындығы анықталды. Темірдің мөлшеріне байланысты оған әртүрлі пішіндегі, яғни параметрлік және магнит тәртіпті күйдегі теміркүрамды агрегаттар қатысуы мүмкін.

УДК 539.19;541.128.13;544.14;544.46

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**ВЗАИМОДЕЙСТВИЕ КАТАЛИТИЧЕСКОЙ СИСТЕМЫ Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>  
С МОЛЕКУЛАМИ-ЗОНДАМИ  
I. ИССЛЕДОВАНИЕ  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> И ИСХОДНОЙ СИСТЕМЫ Fe/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>**

**Аннотация.** Работа является первой частью исследований, посвящённых взаимодействию гетерогенной катализитической системы с адсорбированными молекулами. В ней представлены результаты по исходному

оксиду  $\gamma\text{-Al}_2\text{O}_3$  и системе  $\text{Fe}/\gamma\text{-Al}_2\text{O}_3$  с содержанием железа 0,5; 3; 13 вес.%, полученные с помощью широкого набора физико-химических методов. Система  $\text{Fe}/\gamma\text{-Al}_2\text{O}_3$  была выбрана объектом исследования, поскольку она проявляет каталитическую активность во многих химических процессах и в дальнейшем может быть использована как модельная.

Проведённая работа показала, что в процессе приготовления системы  $\text{Fe}/\gamma\text{-Al}_2\text{O}_3$  методом пропитки возможна модификация структуры носителя. Характер заполнения поверхности носителя железосодержащей фазой существенно зависит от её процентного содержания и может быть многослойным.

Установлено, что система  $\text{Fe}/\gamma\text{-Al}_2\text{O}_3$  содержит железо в форме  $\text{Fe}^{3+}$ . В зависимости от содержания железа, в ней могут присутствовать железосодержащие агрегаты различного размера, как в парамагнитном, так и в магнитоупорядоченном состояниях.

**Ключевые слова:** гетерогенный катализ, физико-химические методы исследования.

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