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RECOVERY OF ZEOLITE WASTE AT PURIFICATION OF NATURAL GASES

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Annotation: *The purpose of this work is to study the degree and structure of contamination of spent zeolites and their effect on adsorption-desorption characteristics in natural gas desulfurization processes, to determine the number and structure of zeolite transport pores and their role in the adsorption process, to develop an optimal method for the recovery of zeolite waste for their secondary use in desulfurization processes.*

Keywords: *adsorption, zeolite waste, desulfurization, natural gas, desorption characteristics.*

Adsorption processes on synthetic zeolites, which provide high selectivity, are widely used for drying and purification of natural gas.

The main studies of the effectiveness of various zeolites for drying and purification from H_2S, CO_2 and NO_2, mercaptans are analyzed. It has been revealed that the most effective adsorbent for drying gas is zeolite Nax, zeolite Naa is the most effective adsorbent for absorbing carbon dioxide from the gas stream.

Modern scientific and technological progress is associated with the constant acceleration of the consumption of natural resources and the development of production.

The Republic of Uzbekistan, having its own potential subsurface resources due to their slow involvement in economic turnover, is forced to import a number of industrial materials and reagents, including expensive synthetic zeolites for gas processing, which are disposed of as waste after operation. Therefore, the issue of extending the service life of imported zeolites, i.e. restoring the original properties of spent zeolites and their secondary use in gas processing is of urgent importance.

The purpose of this work is to study the degree and structure of contamination of spent the zeolite and their effect on adsorption-desorption characteristics in natural gas desulfurization processes, to determine the number and structure of the zeolites transport pores and their role in the adsorption process, to develop an optimal method for the recovery of the zeolites waste for their secondary use in desulfurization processes.In solving the above tasks, the methods of physical chemistry, quantitative and qualitative analysis of pollution products, the theory of redox reactions of the gorenje process, methods of calculating mass-heat exchange processes are used in the work.

The study of the composition of spent zeolites showed that during long-term operation of zeolites in adsorption-desorption cycles at industrial natural gas desulfurization plants, they are contaminated with oxidation-reduction reaction products (hydrocarbon, hydrogen sulfide, carbon dioxide and water vapor) contained in raw gas, which leads to the deposition of these products on the zeolite surface and a sharp narrowing transport pores, and this, in turn, leads to a decrease in the dynamic activity of the zeolite.

The main factor determining the molecular sieve properties is the size of the entrance windows of zeolites, which depends on the location of the oxygen rings of the zeolite and on the number of oxygen atoms in the ring. The size of the entrance window is also influenced by the size of the cation that is part of the zeolite.

The following table shows the results of laboratory studies to determine the degree of contamination of various imported synthetic zeolites, depending on their service life (Table 1.).

Table 1.

Contamination of zeolites depending on the service life

Service life Brand of zeolite	Degree of contamination, in %							
	1 month		12 months		18 months		24 months	
NaA (4A) (USA)	0,135 0,137 0,136	0,136	1,47 1,42 1,45	1,44	2,17 2,20 2,15	2,17	2,94 2,91 2,86	2,9
CaA (5A) (USA)	0,139 0,133 0,136	0,136	1,52 1,46 1,50	1,49	2,25 2,21 2,23	2,23	2,97 3,02 2,94	2,97

The structure of contaminants and their adsorption properties of zeolites were determined by changes in the kinetic and dynamic properties of adsorbents.

It is known that the main characteristic of adsorbents is their absorption capacity, i.e. the degree of gas purification during adsorption. The absorption capacity of the spent zeolites was determined by removing the adsorption isotherms on a device, the principle of operation of which is based on measuring the change in gas pressure during adsorption at a constant volume.

The process of zeolite reduction, oxidation of pollution products, occurs apparently by the following reaction with the release of a significant amount of heat. To carry out this reaction, a sufficiently high temperature is required (400-450^{0}C), but at the same time, due to the thermal effect of the reaction, the temperature of the adsorbent layers can overheat by another 150-200^{0}C.

To prevent such overheating, a reagent was used, which decomposes endothermically under the conditions of zeolite reduction and allows to regulate the temperature of the oxidative reaction to a certain extent. The results show that this method of recovery is considered more effective than the previous ones, and the degree of recovery reaches 100% or more. This is apparently due to a certain expansion of the transport pores due to some dissolution of the zeolite binding materials. However, it should be noted that the mechanical characteristics of zeolite granules deteriorate somewhat. In addition, the recovery technology requires additional costs for ammonium nitrate, distilled water, and the energy costs associated with pre-drying of zeolite will increase.

As is known, zeolites, in addition to their main pores enclosed in the cavities of crystal lattices, have so-called transport pores (capillaries), which play an important role in adsorption processes. The kinetic and dynamic properties of zeolites are mainly determined by these pores. Unfortunately, there are practically no studies in the literature related to the study of the role of transport pores and their effect on the adsorption properties of zeolites.

Generalized results of studying the kinetics of H_2S adsorption on waste (contaminated) and recovered zeolites allows for the first time to experimentally determine the number and size of these transport pores. The rationale for the determination method is a comparison of H_2S adsorption isotherms in spent and recovered zeolites, which confirms our assumptions that as a result of multiple adsorption-desorption cycles of the desulfurization process, only transport pores are polluted.

In addition, in the process of oxidative reduction of spent zeolites, pollution products consisting mainly of carbon (C), hydrogen (H) and sulfur (S), in a coke-like state, interacting with air oxygen, turn into volatile substances (CO_2, SO_2, H_2O and CO) and are removed from the transport pores of the zeolite.

Based on the results of laboratory studies aimed at finding the optimal mode of recovery of zeolite waste and a feasibility study of the recovery method, it was possible to abandon the preliminary screening.

The following table 2 shows the test results.

1. During long-term operation of zeolites in the adsorption–desorption cycles of desulfurization plants, they are contaminated with oxidation-reduction reaction products contained in crude gas (hydrocarbons, hydrogen sulfide, carbon dioxide and water vapor), which leads to a sharp narrowing of transport pores, resulting in a decrease in its dynamic activity.

2. As a result of studying the kinetics of hydrogen sulfide adsorption on spent and recovered zeolites and using the mathematical apparatus of mass transfer theory, a method for determining the structure of transport pores was developed and a change in the dynamic characteristics of zeolites from the narrowing of the capillary size as a result of contamination was revealed.

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