# **CHANGE OF PHYSICO-CHEMICAL PROPERTIES BY MECHANICAL ACTIVATION OF ZEOLITES**

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## **ABSTRACT**

*The study (Navbakhor district of Navoi region) consists of the study of the properties of natural (mining) bentonite mineral raw material to its physical and chemical properties through mechanical activation. X-ray phase and X-ray spectral analysis of the composition of the layer montmorillonite was performed. Mechanical and chemical activation of zeolites to change the surface area and particle size was carried out using a high-power mill AGO-3 and a conical vibrating grinder VKMD-6. The dependence of the mechanical activation of layer montmorillonite on time over 20s, 40s, 80s and 160s proved that it increases the surface area. Using the Karman-Kozeni method, optimal regimes were determined by doubling the mechanical activation time and studying the density, sorption properties, and specific surface area of the zeolite.*

**KEYWORDS:** *Bentonite, Montmorillonite, X-Ray Phase, X-Ray Spectral, Mill, Conical Vibrating Grinder, Karman-Kozeni, Surface Area.*

## **INTRODUCTION**

Currently, there is a growing interest in the development of new environmentally friendly sorbents, retainers and catalysts based on natural clay materials and aluminosilicates. Natural zeolites are a unique mineral with catalytic, adsorption and ion exchange properties, and are relatively inexpensive raw materials compared to synthetic materials. Today, about two hundred different types of zeolites are used, which are divided into inorganic, organic and synthetic types. Due to the good location of the absorbed molecules, zeolites are well-developed mesogamous crystals, they are molecular sieves **[1,2].** Their crystal lattice is formed in the form of a siliconoxygen tetrahedron consisting of four, five or more rings. In some of these tetrahedra, the silicon atom is replaced by aluminum. The resulting crystal lattice nodes are mainly replaced by sodium, calcium, and potassium cations instead of negative charges. As a result of such a structure, zeolites are located in the inner crystalline space, alternating between interconnected mesopoietic

cations into water molecules **[3-7].** The porous mesostructure of zeolites has the best properties: adsorption, molecular sieve, ion exchange and catalytic properties. (Navbakhor district of Navoi region) studied the properties of natural (mining) bentonite mineral raw material by its physical and chemical properties through mechanical activation **[8-12].** The object of study is Navbakhor bentonite clay deposit - in Navbakhor district of Navoi region, on the south-western slope of the South Nurata ridge. It is located 12 km north of Kalqonota village. Con was born in 1998 in A.C. U. Mirzaev and H. Chinqulovs оpened by. The bentonite deposit in Navbahor district has large reserves of natural montmorillonite, which are extracted from open layers.

## **EXPERIMENTAL PART**

Montmorillonite is the most common natural zeolite, whose crystals consist of a threedimensional aluminosilicate-oxygen tetrahedron  $(Si, A)O<sub>4</sub>$ , a mineral with a high volume surface composed of micro, meso, and macro pores.

Chemical formula  $(Na,Ca)_{0,3}(A1,Mg)_{2}[Si_{4}O_{10}](OH)_{2} \cdot nH_{2}O$  [13]. In natural montmorillonite, the molar ratio of  $SiO_2$  /  $Al_2O_3$  is 9-12 and more [14]. Montmorillonite, which belongs to the group of thin-plate zeolites, is very resistant to dehydration and adsorbs H2O and CO<sup>2</sup> well **[15].**



Figure 1. Projections of montmorillonite and montmorillonite structure.

The design model of montmorillonite and montmorillonite structure is shown in Figure 1 **[16- 20].** However, the thermal stability of montmorillonite in air is often around 550-600℃, and the porous diameter of montmorillonite is 0.2 nm **[21].**



Figure 1. Elemental composition and X-ray spectral analysis of the original montmorillonite obtained from the layer.

Mechanical chemical activation of zeolites to change the surface area and particle size was carried out using a high-power mill AGO-3 and a conical vibrating grinder VKMD-6. Grinding in the AGO-3 mill was carried out at intervals of 40 s, 80 s and 160 s - by doubling the crushing time.In VKMD-6, grinding was carried out by increasing the number. Determination of the specific surface area of zeolites by the Karman-Kozeni method showed that with increasing mechanical activation time and crushing number, the specific surface area of the particles increases rapidly and the maximum time of 160 s corresponds to the "8" mode in AGO-3 and VKMD-6. mechanical activation is performed.

#### **RESULTS AND DISCUSSION**

The surfaces of the obtained fine montmorillonite powder particles were examined electronically by microscopy (Figures 2, 3).

The AGO-3 mill showed an electron microscopic photograph of the montmorillonite surface after 40, 80, and 160 seconds of mechanical activation.



MA - 40 seconds

MA - 80 seconds

MA - 160 seconds

Figure 2.in AGO-3 of the zeolite surface in the sample a) 40 s; b) 80 s; c) 160 s; SEM photo in mechanical activation mode



Figure 3. In the "8" dispersion mode of the zeolite surface in VKMD-6: a) the initial sample; c) SEM photo of the sample after grinding

Optimal regimes were determined by doubling the mechanical activation time using the Karman-Kozeni method and studying the zeolite density, gas absorption, and specific surface area.As the mechanical activation time increases, the relative particle size of the powder decreases. Granulometric analysis of zeolite was performed to obtain information on particle size distribution. It can be observed that the particle size of Montmorillonite decreases with increasing fracture. Measurement values depending on the amount of dispersion, equivalent diameter of particles and distribution histograms on the surface morphology of zeolite particles were obtained in VKMD-6 Figure 4.





From the data in Figure 4, the value of the specific surface area of zeolite particles varies depending on the time of mechanical activation.As the mechanical activation time increases, the specific surface area of the particles increases rapidly, and then after reaching a maximum at 160 s, the activation begins to decrease.





Sorption properties are affected by factors such as the degree of crystallinity. Detection of this property was performed for mechanically activated samples. The values obtained for the mechanically activated sample by the following formula are given in Table 2.

$$
k = \frac{(\sum Ii1)}{(\sum Ii2)} \times 100\%
$$

k -crystalline degree,%;

Ii 1 is the maximum intensity of the initial test sample;

Ii 2 is the maximum intensity of the mechanically activated sample.

#### **TABLE 2. THE DEGREE OF CRYSTALLIZATION OF MECHANICALLY ACTIVATED SAMPLE POWDER**



It was found that the samples differ in crystalline level, and the maximum 160 s mode in AGO-3 corresponds to a larger value of this coefficient than in "6" mode in VKMD-8.

It can be said that it has been experimentally determined that the specific surface and the degree of crystallinity are directly proportional to each other, so that the larger the surface, the higher the degree of crystallinity.

Bentonites are not considered pure raw materials, and the main phase, along with montmorillonite, contains a mixture of different minerals, depending on the deposit to be mined.

X-ray phase analysis for bentonites was performed to determine the mineralogical composition. Figure 5 shows X-ray diffractograms of natural bentonites, as well as bentonites fired in an inert argon atmosphere at 550 ℃.



Figure 5. Diffractograms of natural Navbakhor bentonite (1) and sample (2) fired in an inert argon atmosphere at 550℃.

X-ray structural analysis of natural bentonites shows the presence of montmorillonite and  $α$ cristobalite. For the purpose of activation, 550oc burn showed the silcification of bentonites from 17,9 Å to 10 Å of water loss between these layers. Diffraction peaks of X-ray radiation of the studied montmorillonite powder were obtained experimentally according to the mode of mechanical activity.

### **CONCLUSIONS**

The presented dependences indicate the existence of an optimal time and mode of mechanical activation, which experimentally corresponds to the maximum specific surface area and maximum sorption capacity of montmorillonite powder. This suggests that mechanoactivation of montmorillonite is a method that improves its adsorption properties.

During mechanical movements in the AGO-3 mill, the reflection lines from the planes shown in the work disappear on the radiograph for 320 seconds, dispersing the associated particles into the nanoscale state, in which X-rays occur at large angular intervals.It was found that the adsorption properties of zeolites depend on the degree of crystallinity.

For samples with high crystallinity, the adsorption properties will be higher over time than for samples with low crystallinity.

Mechanical activation in the AGO-3 planetary mill is an effective crushing method and a method of increasing the sorption properties of montmorillonite.

X-ray structural analysis of natural bentonites shows the presence of montmorillonite and αcristobalite. As a result of firing for the purpose of activation, a change in the mineralogical composition of bentonites is observed and an illite phase occurs.

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