

HIGHLY EXPRESSED MESOPOROUS SYNTHESIS BASED ON KAOLIN ALUMINOSILICATE

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Abstract. A method for obtaining a highly dispersed mesoporous artificially obtained aluminosilicate X based on the crystallization of a natural aluminosilicate with a highly active methacaoline structure in a solution of sodium metasilicate at a temperature of 96-98 ° C. Calculations show that at a temperature of 30 ° C and an initial mass or volume of sodium oxide of 120 g/l, the maximum amount of dissolved aluminum and silicon (IV) oxides is 2.4 and 20, respectively, of their content in methacaoline with high activity. Mesoporous artificially obtained aluminosilicate X-components of high hardness and phase purity are converted from reaction mixtures into solids: (2,3-2,6) Na₂O*Al₂O₃*(3,0-3,5) SiO₂*(60-80) H₂O. The distribution of pores by surface area and size was determined by low-temperature nitrogen desorption in an automatic adsorbent "ASAB 2010". Sedimentation analysis was performed in various dispersion media in water and water-glycerin mixture using the Oden method. X-ray phase analysis (CoKa radiation) was performed on a DRON-4 diffractometer with a cobalt X-ray tube. The PDF-2 database of the International Diffraction Data Center (JCPDS, 1999) was used to analyze diffractograms. The parameters of the porous structure of the samples were determined by low-temperature nitrogen desorption on a Quanchrome NOVA analyzer (USA).

The aim of the work is the synthesis of highly dispersed mesoporous aluminosilicates based on kaolin.

Keywords: kaolin, NaX aluminosilicate, crystallization, Karnab ota kaolin.

INTRODUCTION

With the growth of fuel and energy consumption in developed countries and the growth of the economy and population in developing countries, the global demand for energy is constantly increasing. In the modern world, the availability and high demand for fuel and energy resources, their uninterrupted supply to consumers and ensuring their effective use by consumers are becoming the main guidelines for the development of energy strategies in different countries of the world [1-6].

The presence of hydrogen sulfide in hydrocarbons causes a number of problems: the danger of corrosion destroys the development technique, pollutes the atmosphere as a result of combustion, poses a great danger to living organisms and biosystems when burned in furnaces and flares, pollutes the environment. , disrupts the energy balance of the Earth, biological, biochemical cycle breaks [7-9]. In addition, if hydrocarbon raw materials are involved in chemical processes, hydrogen sulfide will disrupt the technological process, since it is very aggressive and forms harmful compounds in combination with catalysts. Hydrogen sulfide and sulfur (IV) oxide pose not only a technological risk, but also pose a great risk to living organisms and the environment

during extraction and operation or during transportation. Hydrogen sulfide (H₂S) is a colorless gas with a characteristic odor. It is heavier than air, its density under normal conditions is 1.5092 kg/m³ (relative humidity 1.17), so it accumulates in places with low air exchange. H₂S is highly flammable (ignition temperature 2460°C) and explosive when mixed with air. When mixed with air, the flammability limit (lower and upper limit) under normal conditions is 4.3-45.5% by weight. Therefore, hydrogen sulfide is prone to ignition and explosion in the air, and if it is mixed with a combustible gas, the explosion will be strong. The self-ignition temperature of hydrogen sulfide is 2900C. The effect of hydrogen sulfide on the human body depends on its concentration in the environment [10-16]. It is known that in the environment, it is not hydrogen sulfide that has a negative effect on living organisms, but sulfur oxide (IV), the product of its combustion. Sulfur oxide (IV) SO₂ is a colorless burnt sulfur with a pungent odor. Sulfur oxide (IV) under normal conditions has a boiling point of minus 100C, a heat of evaporation of 24.94 kJ/kmol, a melting point of -75.50 C and a melting heat of 7,400 kJ/kmol. Currently, as a result of the development of industry and human activities, the emission of sulfur compounds into the atmosphere amounts to several million tons, and when burning solid, liquid and gaseous fuels containing sulfur, a lot of sulfur dioxide (SO₂) is released into the atmosphere [17-21]. At this time, about two kilograms of sulfur dioxide are obtained from one kilogram of hydrogen sulfide. Sulfur dioxide is the most polluting substance, it returns to earth in the form of acid rain after a fire. SO₂ has a settling rate of 0.001-0.02 m/s, which means that it fell to the ground faster than other elements, such as nitric oxide, even small particles (in aerosols).

Sulfur dioxide in the atmosphere has a bad effect on the body, plants and living beings, and also causes corrosion of metals. Land subsidence affects soil composition, vegetation, reduces fertility, negatively affects flora and fauna, causes desiccation of forests, pollutes water. More sulfur dioxide is formed when burning fuel oil, coal, mine flares, gas fuel, sulfur-containing fuels. The concentration of sulfur dioxide in the exhaust gases is mainly proportional to the sulfur content in the fuel. Among sulfur compounds, sulfur anhydride is considered the main environmental pollutant, since it emits a large amount of toxic gas into the atmosphere, which causes great harm to living organisms and the environment [22-25]. When hydrocarbons ignite without pretreatment, the environment is polluted with sulfur anhydride, which spreads within a radius of 8-12 km from this place. Therefore, since hydrogen sulfide and its compounds emit an aggressive poisonous gas during combustion, restrictions on the amount of hydrogen sulfide in the air are introduced in all countries of the world [26-28].

In organic and petrochemical synthesis, the interest of world scientists is the production of ethylene hydrocarbons from methane, synthesis gas and methanol and dimethyl ether based on it, vinyl acetate and acetone from acetylene, as well as the creation of sorbents and zeolite catalysts with high efficiency in these processes [30-47]. Demand for the quality of hydrocarbon products is growing in many countries, and this demand is increasingly tightly controlled. Quality control of gas supplied to the public and gas supplied to the chemical industry (especially in the synthesis of hydrogen and gas) is becoming increasingly stringent [48-51].

THE EXPERIMENTAL PART

To determine their chemical and physico-chemical characteristics, 100 g of granule samples were placed in a 250 cm³ glass flask and 150 cm³ of distilled water was added. The flask was stirred for 24 hours in the AVU-6 apparatus at 120 rpm. After drying, the adsorbent was passed through a 0.5 and 0.25 mm sieve and the mechanical and physico-chemical characteristics of the samples passed through a 0.5 mm sieve and remained on a 0.25 mm sieve were studied. Before acid treatment, the soils are crushed to a size of 0.08 mm. 40 ml of heated H₂SO₄ was added to 10 g of crushed soil and heated with stirring in a water bath. After treatment, the soil was filtered

through a paper filter on a Buchner funnel and washed with distilled water in the pH range = 5.4-5.7. Then the soil, together with the filter paper, was dried in a drying cabinet at a temperature of 120 ° C for 5 hours. The distribution of pores by surface area and size was determined by low-temperature nitrogen desorption in an automatic adsorbometer "ASAB 2010". Sedimentation analysis was performed in various dispersion media in water and water-glycerin mixture using the Oden method.

X-ray phase analysis (CoKa radiation) was performed on a DRON-4 diffractometer with a cobalt X-ray tube. The PDF-2 database of the International Diffraction Data Center (JCPDS, 1999) was used to analyze diffractograms. The parameters of the porous structure of the samples were determined by low-temperature nitrogen desorption on a Quanchrome NOVA analyzer (USA). Before measurement, each sample was degassed in vacuum at a temperature of 250 ° C for 2 hours. The Brunauer-Emmett-Taylor (BET) method was used to determine the relative surface area of solid samples. The following BET equation was used in this method:

$$\frac{1}{W \cdot \left(\frac{P_0}{P} - 1\right)} = \frac{1}{W_m \cdot C} + \frac{C - 1}{W_m \cdot C} \cdot \frac{P}{P_0}$$

where WP/P_0 is the mass of the gas adsorbed at relative pressure, is the mass of the substance adsorbed on the surface covered with the forming monolayer; C —adsorbents is the BET constant, which indicates the magnitude of the adsorbate interaction, which is the dependence of the adsorption energy of the first adsorption layer W_m .

The Barrett-Joyner-Halenda (BJH) method was used to determine the pore size and size distribution. Desorption and adsorption areas of the isotherm in the pressure range 0.967-0.4 P/P_0 were used as initial data in the calculations.

To determine the density of the pile, 500 g of sorbent was taken and kept at a temperature of 600C for 2 hours in a drying cabinet. 400 g of the dried sample was taken, placed in a measuring cylinder with a capacity of 500 ml and its volume V_1 was measured. Then, with light taps on the bottom of the cylinder, the sorbent was compacted to a constant level and the volume V_2 was measured again.

The bulk density of the sorbent was determined by the following formula:

$$\gamma_1 = \frac{P}{V_1}; \gamma_2 = \frac{P}{V_2} \text{ g/cm}^3$$

where is the mass of the P –sorbent; , is the bulk density before and after compaction of the sorbent, g/cm^3 . $\gamma_1 \gamma_2$

THE RESULTS OF THE EXPERIMENT AND THEIR DISCUSSION

Mesoporous, artificially produced aluminosilicates cannot be cured directly from kaolin extracted at the Karnab Ota mine. Therefore, kaolin obtained at the Karnab Ota mine undergoes thermal dehydration, which leads to the formation of metakaolin, which is a reactive raw material for the synthesis of mesoporous, artificially obtained aluminosilicates. Based on kaolin obtained at the Karnab Ota mine, several methods have been developed in our country and abroad for the production of granular (granules) mesoporous artificially obtained aluminosilicates A without binders. The inclusion of only at least 30% of finely dispersed mesoporous artificially obtained aluminosilicate A in the composition of the initial granules (granules) makes it possible to obtain a product with a degree of crystallization close to 100% after crystallization, which indicates the absence of a mixture of sodalite phases. Considering that the synthesis of highly dispersed mesoporous, artificially obtained aluminosilicate from kaolin obtained at the Karnab Ota mine is impossible without the introduction of granular (granular) mesoporous, artificially obtained aluminosilicate A without binders, silicon as the main Source (IV), we proposed to develop a

method for obtaining artificially obtained aluminosilicate A with high dispersion of mesoporosity using metacaolin with high activity of aluminum oxide.

The regularities of obtaining and forming artificially obtained highly dispersed mesoporous NaA aluminosilicates from $Al_2Si_2O_7$ have been studied.

However, mesoporous synthetic aluminosilicate X has a lower average volume of solids than particles of highly active methacaolin. Thus, a highly dispersed mesoporous, artificially obtained aluminosilicate X was developed, based on the crystallization of a natural aluminosilicate with a highly active methacaoline structure in a solution of sodium metasilicate at 96-98 ° C (Fig. 1). Mesoporous artificially obtained aluminosilicate X components of high hardness and phase purity are converted from reaction mixtures into solids: $(2,3-2,6)Na_2O \cdot Al_2O_3 \cdot (3,0-3,5)SiO_2 \cdot (60-80)H_2O$

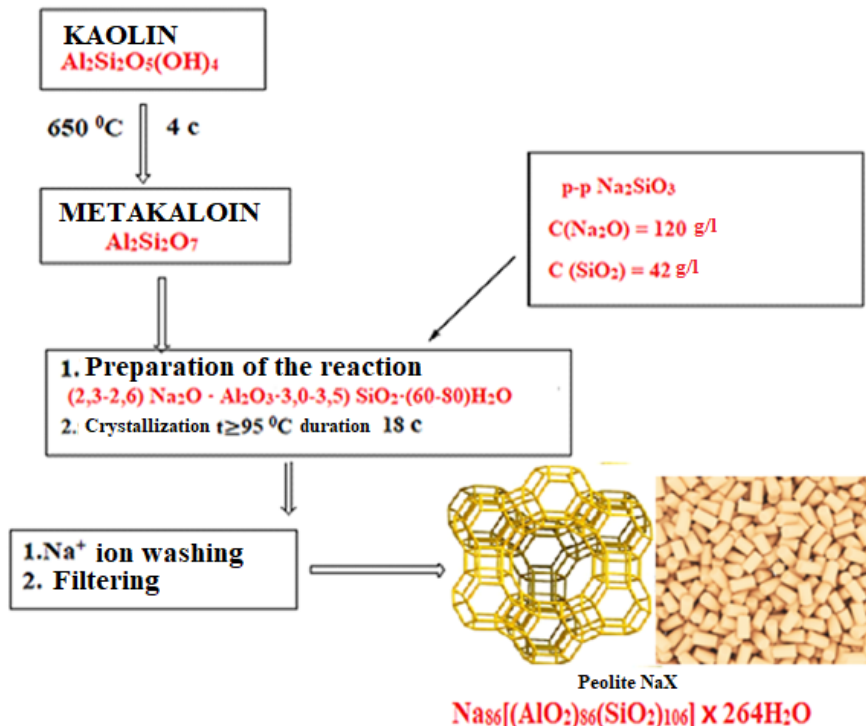


Figure 1. Synthesis of highly dispersed mesoporous artificial NaX aluminosilicate from kaolin obtained at the Karnab Ota mine

In the process of crystallization of highly active methacaolin into mesoporous, artificially obtained aluminosilicate X, due to the difference in chemical composition, mass transfer between the liquid and solid phases of the reaction mixture plays a more important role than during hydrogel crystallization. Simultaneously with the transition of sodium to the solid phase, aluminum oxide appears in the solution, the amount of which per unit mass or volume gradually increases throughout the daily treatment, and $C(Al_2O_3) = 3.2 / l$. Given the presence of metasilicate ions in the initial solution, it can be expected that they will be added to the solid phase at the early stages of thermochemical treatment. At the same time, during the first 8 hours, the amount of mass or unit volume of silicon in the liquid phase increases, and $C(SiO_2) = 64.4 \text{ g/l}$, and then begins to decrease. When the amount of sodium oxide in the initial unit of mass or volume of the crystallization solution increases to 230 g/l, the amount of aluminum and silicon (IV) oxides per unit of mass or volume increases by 4.4 and 66.4 g/l, respectively. Calculations show that at a temperature of 30 ° C and an initial mass or volume of sodium oxide of 120 g/l, the maximum amount of dissolved aluminum and silicon (IV) oxides is 2.4 and 20, respectively, of their content in methacaoline. with high activity. The composition of the liquid and solid phases of the reaction mixture is shown in Table 1.

Table 1

Na₂O/ in the liquid and solid phases of the reaction mass after 8 hours of treatment at 30 °C in solutions of sodium metasilicate with different amounts of sodium oxide per unit volume Al₂O₃/SiO₂ ratio

The amount of solution in units of mass or volume, g/l		Molar ratio in the liquid phase			Molar ratio in the solid phase		
Na ₂ O	SiO ₂	Na ₂ O	Al ₂ O ₃	SiO ₂	Na ₂ O	Al ₂ O ₃	SiO ₂
120	42	85,4	1.0	54,7	0,2	1.0	1,9
150	42	88,8	1.0	41,6	0,3	1.0	1,8
230	42	114,3	1.0	38,9	0,6	1.0	1,8

As can be seen from the table above, the composition of the phases differs from each other and from the mesoporous, artificially obtained NaX aluminosilicate. At 30 °C, no mesoporous synthetic aluminosilicate is formed for 24 hours. The accumulation of amorphous sodium aluminosilicate occurs in the solid phase.

It should be noted that the amount per unit of mass or volume is significantly reduced compared to 30 °C. This indicates a denser mass transfer between the highly active methacaolin and the sodium metasilicate solution. In the liquid phase of the reaction mass for their oxides, the amount of silica (IV) oxide per unit mass or volume of aluminum oxide increases during the first 8 hours at 60 °C and reaches a maximum level. They decrease with a further increase in processing time. Calculations show that at a temperature of 60 °C and the amount of sodium oxide in the initial unit of mass or volume in a crystallization solution of 120 g / l, the largest number of aluminum oxides and silicon oxide (IV) by the number of oxides received, the solution, respectively, in its composition of metakaolin with high activity is 3.4 and 13.1%.

The amount of silicon (IV) aluminum oxide per unit mass or volume in the liquid phase increases during the first 4 hours of treatment at 98 °C and reaches the highest values in two cases when 120 and 155 g/l per unit mass or volume of Na₂O solutions. They decrease with a further increase in processing time. If Na₂O is a solution of sodium metasilicate with a mass or volume of 230 g / l, then there is a gradual increase in the amount of aluminum oxide and silicon oxide(IV) in the solution. As the results of X-ray phase analysis showed, the degree of hardness was high. Na₆(AlSiO₄)₆•8 H₂O forms a phase with a structural formula. The data obtained show that at a temperature of 98 °C and the amount of sodium oxide in the initial mass or volume unit of 120 g / l in the crystallization solution, the largest amounts of aluminum oxide and silicon oxide (IV) pass into the crystallization solution. the solution is 2.6 and 9.1% of their composition. These values increase to 4.2 and 15.5% with an increase in the amount of Na₂O per unit mass or volume in solution to 155 g/l.

It should be noted that at high temperatures, the amount of sodium per unit mass or volume decreases more than at low temperatures. This fact indicates a great depth of interaction of metakaolin with high activity and solution. The amount of silica (IV) alumina per unit mass or volume in the liquid phase increases during the first 4-8 hours of processing and reaches a maximum level. It should be noted that at 30 °C, the amount of silicon oxide (IV) per unit mass or volume of alumina is greater than at 60 and 98 °C.

The amount of highly active methacaolin Na₂O per unit mass or volume is from 50.0 to 150.0 g/l and SiO₂ in aqueous solutions, sodium metasilicate with a unit mass or volume of 90.5 g/l has converted to a solid state. The PC compositional associations varied within the following limits (2.0-2.2) Na₂O * Al₂O₃·(6,0-6,5)·(150-160) H₂O is mainly from solutions of salts and sodium metasilicate formed by the action of alkali on freshly precipitated aluminum hydroxide.

The same structural associations were used as in the synthesis of a highly dispersed mesoporous artificially obtained aluminosilicate Y.

CONCLUSION

Thus, a method has been developed for the production of a highly dispersed mesoporous artificially obtained aluminosilicate X, based on the crystallization of a natural aluminosilicate with a highly active metakaolin structure in a solution of sodium metasilicate at 96-98 ° C. Calculations show that at a temperature of 30 ° C and an initial mass or volume of sodium oxide of 120 g/l, the maximum amount of dissolved aluminum and silicon (IV) oxides is 2.4 and 20, respectively, of their content in methacoline. with high activity. Mesoporous artificially obtained aluminosilicate X-components of high hardness and phase purity are converted from reaction mixtures into solids: $(2,3-2,6)Na_2O \cdot Al_2O_3 \cdot (3,0-3,5)SiO_2 \cdot (60-80)H_2O$

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