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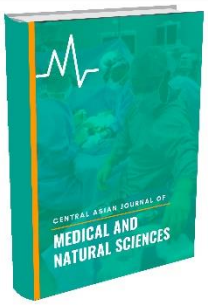

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SYNTHESIS OF MESOPOROUS Al_2O_3 AND STUDY OF ITS SORPTION PROPERTIES

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ABSTRACT: A mesoporous Al_2O_3 sorbent was synthesized using sol-gel technology. The sorbent's phase composition was analyzed via X-ray diffractometry (XRD), while its surface morphology was studied using scanning electron microscopy methods. Textural characteristics were also investigated through the adsorption of toluene vapors on the sorbents with the help of Mak-Ben-Bakra's sensitive quartz spiral device. The specific surface area (SBET) of sorbents obtained at different temperatures was found to be 684,6 ± 10 m^2/g, with a volume of pores (Vs) of 0,56 to 0,82 cm^3/g and an average pore diameter of 12,4 ± 0,05 nm. X-ray microanalysis of the sorbents confirmed that their chemical composition corresponds to γ- Al_2O_3.

KEYWORDS: sol-gel, mesoporous sorbent, diffractometry, specific surface area, pore volume.

INTRODUCTION

Currently, aluminum oxide and composite materials based on it find wide application in various industries [1-2]. This material is particularly useful in catalysis, oil refining as a sorbent, wastewater treatment, the production of chemical sensors, solving environmental problems, metallurgy, mechanical engineering, obtaining ceramic composite materials, and in all fields of electronics [3-7]. The importance of aluminum oxide in these applications cannot be overstated.

There are various types of powders and dispersions of Al_2O_3 nanoparticles that exhibit differences in terms of molecular and crystal structure, particle size, relative surface area, and physicochemical properties. Upon high-temperature calcination of γ-Al_2O_3, two structurally distinct polymorphic types, δ-Al_2O_3 and θ-Al_2O_3, are formed. In δ-Al_2O_3, the Al^{3+} cation is located at the ends of the octahedron, while in θ-Al_2O_3, it is situated at the ends of the tetrahedron.

Al_2O_3 and mesoporous nanomaterials derived from it can be synthesized using several methods, such as solution-based approaches, vaporization from the gas phase, hydrothermal treatment, microwave heating, and template synthesis based on a solid template [8-12]. Among these methods, the sol-gel

technology is the most convenient for obtaining pure porous aluminum oxide due to its simplicity of equipment, reproducibility, ecological safety, and cost-effectiveness of the resulting products. Sol-gel synthesis involves the incorporation of various monomers with different functional groups, polymers, and metal oxides into the reaction system, making it a versatile method. Additionally, it utilizes a single solvent for all reagents and allows for hydrolytic reactions to occur under mild conditions [13-15].

To investigate the use of mesoporous Al_2O_3 in wastewater treatment, this research work involved the synthesis of the material using water-soluble crystalline hydrates of aluminum and cationic surfactants.

EXPERIMENTAL

Materials and methods

In the synthesis of mesoporous sorbents, γ- Al_2O_3 sols were obtained from $AlCl_3 \cdot 6H_2O$ crystal hydrate, and cetyltrimethylammonium chloride ($C_{19}H_{42}ClN$) was used as a template for mesoporous structure control (purity >98,7%, Jinan Xinggao Chemical Technology Co., Ltd, China). A 0,01M solution of carbamide and NH_4OH (K_d=1,76·10^{-5}, pH=10,3) was used to provide a hydrolysis catalyst and a basic environment. Recycled ethanol (purity >96.2%) was used as a solvent. During the synthesis, the dissolution medium was monitored using a Mettler Toledo FP-20 pH meter.

To study the effect of temperature on the specific surface area (S_{BET}, m^2/g), pore volume (V, cm^3/g), and their average diameter (D, nm) of the obtained sorbents, the synthesis process was carried out at a temperature of 50°C. The textural characteristics of sorbents were studied by the adsorption of toluene vapors in the sensitive quartz spiral device of Mak-ben-Bakra.

To study the surface morphology of the sorbents, SEM was performed using a scanning electron microscope EVO MA 10 (Carl Zeiss, Germany), and the composition of the elements was analyzed using a detector (EDS Aztec Energy Advanced X-Act, Oxford Instruments) that was additionally connected to the microscope.

Synthesis of γ-Al_2O_3 sorbents

The synthesis of mesoporous γ- **Al_2O_3** was carried out using the following procedure first, 1,4 g of cetyltrimethylammonium chloride was dissolved in 23 ml of alcohol. Then, 5 g of $AlCl_3 \cdot 6H_2O$ dissolved in 20 ml of water and 0,7 g of urea were added dropwise to the mixture. The resulting solution was placed in a thermostat and mixed for 30 minutes at an appropriate temperature of 50°C. Then, 8 ml of 0,01 M NH_4OH solution was added dropwise to the solution and stirred until a white suspension was formed. The synthesis was carried out for 3 hours for the resulting solution to become a complete gel. The obtained gel sample was washed several times with distilled water to remove additional products, filtered, and dried gel sample was placed in an oven and heated at 105°C for 6 hours until it was completely dry. After drying, the sample was calcined in a furnace at 550°C for 5 hours to obtain the mesoporous γ-Al_2O_3 sorbent.

RESULTS AND DISCUSSION

The isotherms resulting from the sorption of toluene vapors on γ-Al_2O_3 sorbents, which were obtained at a temperature of 50°C, are shown in Figure 1.

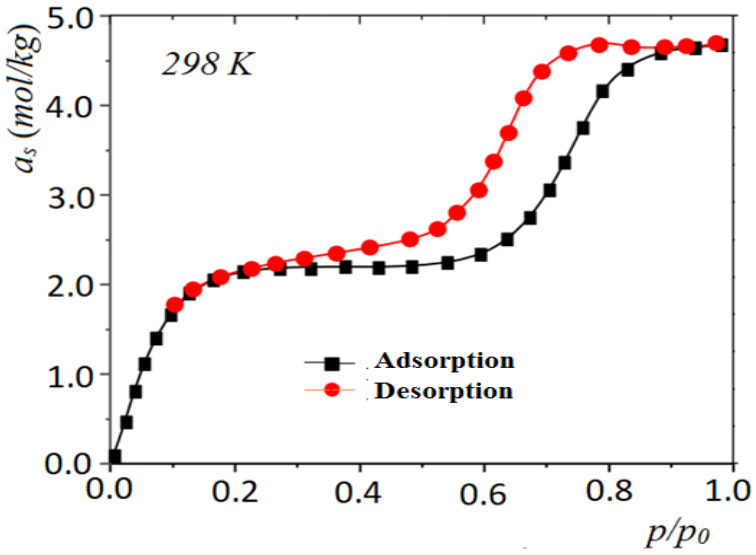

Figure 1. Shows the adsorption isotherms of toluene vapor on the synthesized sorbent at 50°C.

The adsorption lines rise sharply up to a relative pressure of p/p_0=0,4 and approach saturation at p/p_0=0,9, indicating efficient toluene capture. The range of relative pressure p/p_0=0,4÷0,8 shows a hysteresis loop resulting from capillary condensation and the combination of adsorption and desorption lines. The sorbent sample obtained at 50°C consists of mesopores, and the isotherm's shape suggests that it belongs to type IV according to the IUPAC classification.

Using the values obtained in the sorption isotherms association, the saturation of mesopores and the specific surface area of sorbents (S_{BET}, m^2/g), the average diameter of pores (D, nm), the monolayer capacity of sorbents (a_m, mol/kg) and saturation adsorptions (a_s, mol) using the BET isotherm models /kg) was calculated (Table 1).

Table 1. Shows the textural characteristics of the synthesized γ-Al_2O_3 sorbent obtained at different temperatures.

Adsorbent	S_{BET}, m^2/g	a_m, mol/kg	a_s, mol/kg	D, nm
γ-Al_2O_3	684,6±10	2,6±0,2	6,2±0,8	12,4±0,05

From the table, it can be seen that 41,9% of toluene vapors are sorbed to monolayers.

Based on the values obtained from the adsorption of toluene vapors, the saturation adsorption volumes (V_s) of the sorbents, the volume of micropores (W_0), the volume of mesopores (W_{mes}) and the saturation volume (V_s) of toluene vapors were determined (Table 2).

Table 2 . Shows the size of pores and their distribution on the surface in γ-Al_2O_3 sorbents

Adsorbent	$W_0 \cdot 10^3$, sm^3/g	$W_{me} \cdot 10^3$, sm^3/g	$V_s \cdot 10^3$, sm^3/g
γ-Al_2O_3	0,424 ± 0,05	0,852 ± 0,06	1,276 ± 0,10

Studying the sorbent surface morphology using SEM confirmed that its textural characteristics correspond to the above values (Figure 2).

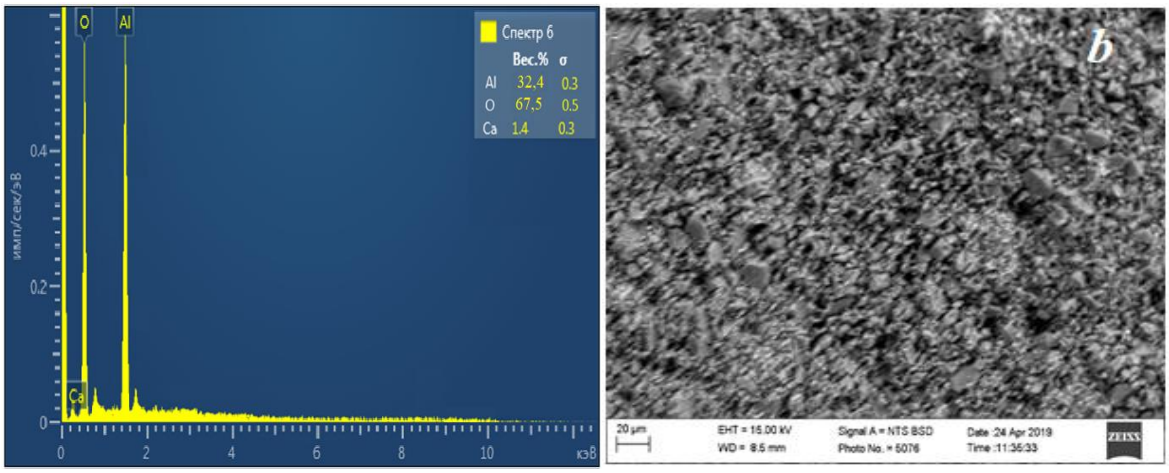

Figure 2. Shows EDS and SEM images of the sorbent

From the picture, the analysis results of individual areas of the surface showed that the composition of the sorbent consists only of Al and O, and there are no additional components in it. It was determined that the composition of Corbent element consists of 32,4±0,2% Al, 67,3±0,6% O by mass.

CONCLUSIONS

Mesoporous Al_2O_3 sorbent was synthesized based on sol-gel technology. Their textural characteristics were studied through the adsorption of toluene vapors on sorbents using the sensitive quartz spiral device of Mac-Ben-Bakra. According to it, the specific surface area (SBET) of sorbents obtained at different temperatures is 684,6·10 m^2/g, the volume of pores (V_s) is 0,56÷0,82 cm^3/g, and the average diameter of pores It was found to be 12,4±0,05 nm. X-ray microanalysis of the sorbents proved that their chemical composition corresponds to γ-Al_2O_3.

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