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SCIENTIFIC STUDY OF THE PHYSICOCHEMICAL PROPERTIES AND THERMAL TRANSFORMATIONS OF USED ZEOLITES

Sultonov Sadulla [1], Kucharov Azizbek [1,2], Yusupov Farkhod [1], Dusmatova Anzirat [2], Toshboboyeva Ra'no [2]

[1] Institute of General and Inorganic Chemistry of the Academy of Sciences of Uzbekistan. Tashkent Uzbekistan

[2] Pharmaceutical Education and Research Institute. Yunusobod District, Tashkent, Uzbekistan

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Abstract

This study investigates the properties of granules prepared using various binding solutions and explores the effect of adding combustible materials and metal oxides to catalysts. Electrolyte solutions significantly increased the specific surface area and total pore volume of the granules, while combustible materials like cellulose decreased density and mechanical strength. A specific amount of calcium oxide improved the specific surface area; however, exceeding the optimal content reduced strength. The addition of calcium hydroxide suspension yielded better results. The optimal composition was identified in catalysts containing calcium oxide and magnesium oxide. Thermal analysis confirmed phase transformations occurring at different temperatures, contributing valuable insights into the material behavior and catalytic properties.

Keywords: *Zeolite, processing, physicochemical properties, thermal analysis, metal oxides, catalyst, surface area, porosity, active components, thermal transformations*

Introduction

Zeolites, with a density range of 2––2.3 g/cm³ (Derouane, Eric G., et al., 1981), play vital roles in adsorption, catalysis, and ion exchange due to their physicochemical properties. Thermal transformations, crucial for stability and activity, significantly impact their performance (Cruciani, Giuseppe. 2006). Current research emphasizes improving the structure, surface area, and pore volume of zeolites to optimize functionality under diverse conditions (Yusupov, F., & Khursandov, B., 2024). For example, clinoptilolite demonstrates a surface area of 300––400 m²/g (Temirov G. et al., 2023), showcasing its adsorption potential. Studies show that modifying zeolites, such as adding calcium oxide, further enhances specific surface area and catalytic efficiency (Khursandov, B. Sh., Kucharov, A. A. U., & Yusupov, F. M., 2022).

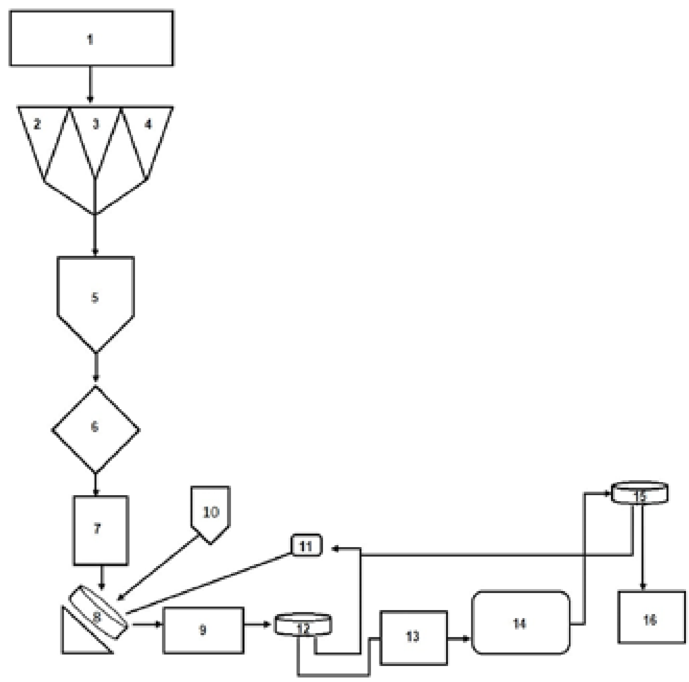
This study aims to investigate the physicochemical properties and thermal behavior of zeolites, focusing on phase transformations and heat resistance (Yusupov, F.M., Mamanazarov, M.M.U., Kucharov, A.A.U., & Saidobbozov, S.Sh., 2020). Enhancements in catalytic and adsorption activities ensure extended application in petrochemistry, environmental protection, and chemical synthesis (Yusupov Farkhod, Azizjon Qurbonov, Kucharov Azizbek, Yodgorov Normahmad, 2025). Results contribute to increasing zeolite efficiency and service life, offering economic benefits alongside ecological impact (Yusupov, F., Kucharov, A., Baymatova, G., Shukurullayev, B., & Yuldashev, R., 2023). Improvements in surface area and pore volume through structural optimization highlight the significance of zeolites in advanced scientific applications (Kucharov, Azizbek, et al., 2025).

Research method

The physicochemical properties of the produced granules can vary widely depending on several factors (Xursandov, Bobomurod, et al., 2024). For instance, the chemical composition, moisture content, particle size distribution, and physicochemical properties of the raw material significantly influence the granules' characteristics (Piccione, Patrick M., et al., 2000). Other factors such as the quantity and ratio of powder and binding solution used in the granulation process, the nature of the binding solution, as well as the granulator's angle of inclination and rotational speed, also play a crucial role (Kucharov, Azizbek, et al., 2021).

For the preparation of catalyst samples, aluminum oxide waste, previously utilized as an adsorbent, was used as the primary raw material (Ozin, Geoffrey A., Alex Kuperman, and Andreas Stein. 1989). Additionally, active components such as CaO, MgO, TiO_2, and V_2O_5 were employed. These oxides were incorporated in the initial stage as powders (e.g., CaO, MgO, TiO_2) or introduced in solution form (e.g., $Ca(OH)_2$, $Mg(OH)_2$, NH_4VO_4) either during the granulation step with the binding solution or by impregnating the final granules with their respective solutions (Qurbonov, Azizjon, Azizbek Kucharov, and Farxod Yusupov, 2024).

Figure 1. *Technological Process Scheme for Producing Zeolite of Sb-14 Brand*



1–2,3,4 – Collecting hoppers; 5 – Dispenser; 6 – Disintegrator; 7 – Collecting dispenser; 8 – Pan granulator; 9 – Reactor for physicochemical treatment; 10 – Reactor for binding solution; 11 – Mill; 12, 15 – Sieves; 13 – Container for active component impregnation; 14 – Furnace; 16 – Storage

Initially, raw materials were crushed to 100 μm. More than 50% of the crushed sample consisted of fractions under 70 μm. The crushed raw materials and binding solution were fed into a rotating pan granulator at a speed of 15–20 rpm, with the granulator's angle of inclination set between 40–60°. Granulation time varied from 15 to 45 minutes. The binding solution was added at 30% of the initial raw material powder weight. The raw material powder was supplied at a rate of 160–180 g/min, while the binding solution was added at 60–80 ml/min. Water, catalyst active component solutions, and NaOH solutions were used as binding agents. Spherical granules with 25–30% moisture content were obtained from the granulator and subjected to physicochemical processing.

The granules were treated with water vapor at 80–90 °C for 2 hours, then sieved and dried at 110–120 °C for 15–20 hours. Active components were impregnated into the dried granules, followed by thermal treatment at 420–550 °C. Heating was conducted at a rate of 70 °C/hour until the desired temperature was reached, and thermal processing continued for 4 hours at that temperature.

Result and discussion

Properties of granules prepared using different binding solutions are summarized in Table 1. The use of electrolyte solutions as binders significantly increased both the specific surface area and total pore volume of the granules. When NaOH and H_3BO_3 were employed as binding agents, the mechanical strength of the granules improved, whereas the use of ethanol led to a slight decrease in strength. Electrolyte-based binders were found to have a notable effect on pore structure. Specifically, the addition of NaOH resulted in an increased number of macropores, while ethanol promoted the formation of micropores and reduced the presence of macropores. In contrast, the inclusion of boric acid had minimal impact on overall pore volume.

Table 1. *The effect of adding electrolytes to the binder solution on the properties of granules*

Binder Solution	H_2O	10% NaOH	10% C_2H_5OH	10% H_3BO_3
Micropores, cm³/g	0.05	0.18	0.28	0.08
Macropores, cm³/g	0.26	0.31	0.16	0.28
Total pore volume, cm³/g	0.31	0.49	0.44	0.39
Density, g/cm³	0.694	0.722	0.697	0.719
Mechanical strength, MPa	4.5	6.2	4.1	5.2

It was found that the addition of various combustible materials to the raw material had different effects on the properties of the granules. The addition of combustible materials significantly decreased the mechanical strength and density of the granules. The inclusion of combustible materials increased the total pore volume, mainly due to an increase in the number of macro- and mesopores. It was also found that the amount and particle size of the combustible materials affected the properties of the granules (Table 2).

Table 2. *Physical and mechanical properties of granules prepared with combustible materials*

Binder Solution	Without additives	Cellulose 8%	Coal 8%	Wood shavings 8%
Micropores, cm³/g	0.05	0.06	0.10	0.07
Macropores, cm³/g	0.27	0.27	0.28	0.35
Total pore volume, cm³/g	0.32	0.38	0.40	0.41
Specific surface area, m²/g	290	240	265	285
Density, g/cm³	0.712	0.676	0.655	0.633
Mechanical strength, MPa	5.7	4.2	3.9	3.3

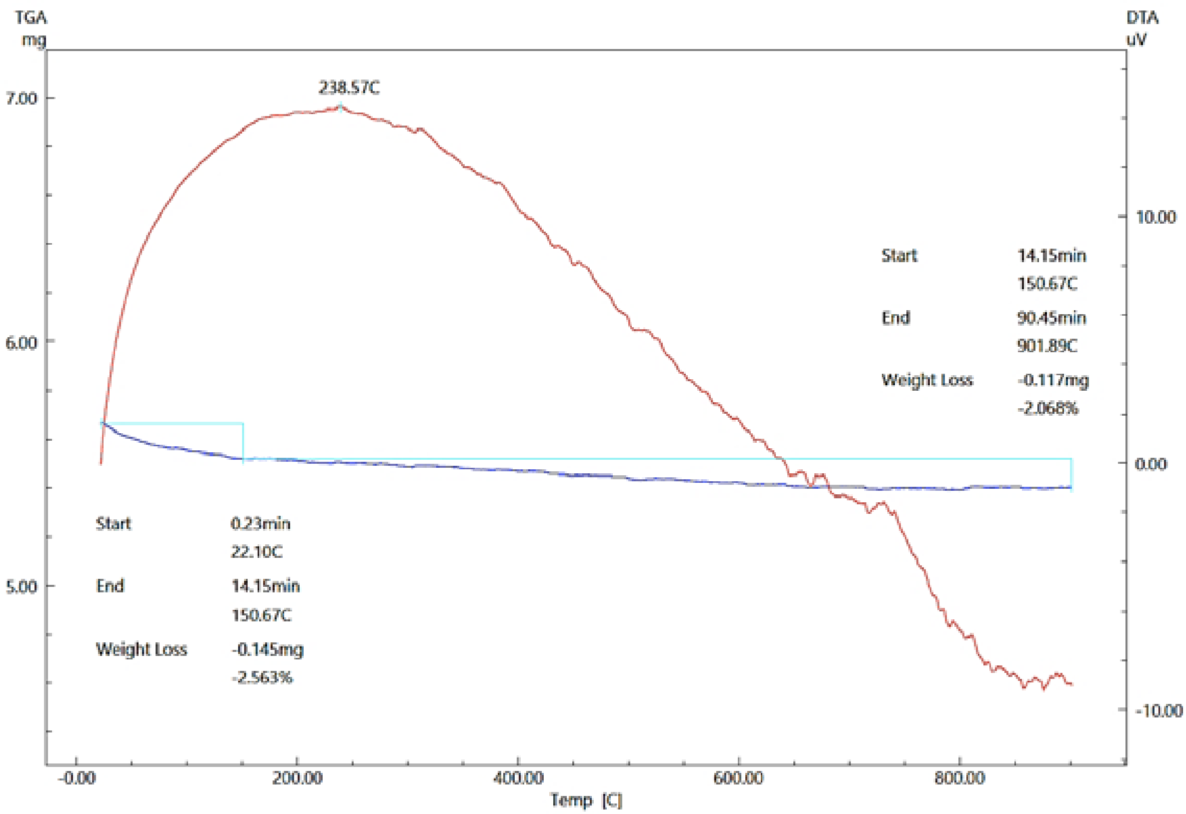
During granule preparation, pure aluminum oxide was also used together with SHKM (adsorbent) waste. Aluminum oxide and SHKM waste were mixed in different ratios to prepare the granules. The physical and mechanical properties of the samples were studied. The results shown in Table 3 demonstrate that the addition of pure aluminum oxide to the composition improved the properties of the granules.

Table 3. *Physical and mechanical properties of granules prepared from different ratios of pure aluminum oxide and adsorbent waste*

Ratio	10/90	20/80	30/70	40/60
Density, g/cm³	0.694	0.705	0.689	0.704
Mechanical strength, MPa	4.4	4.9	5.8	6.1
Specific surface area, m²/g	280	290	310	315
Total pore volume, cm³/g	0.31	0.35	0.41	0.42

The TG, DTG, and DSC curves of the obtained sample are shown in Figure 2. According to the literature (Yusupov, F., & Khursandov, B., 2024), the endothermic effect observed at low temperatures indicates the release of adsorbed water from the sample. The second endothermic effect observed at 250–300 °C indicates the formation of boehmite and the low-temperature aluminum oxide phase – η-Al_2O_3. The endothermic effect at 500–700 °C is associated with the release of water from boehmite-structured aluminum hydroxide and the formation of γ-Al_2O_3.

Figure 2. *Thermal analysis of a sample obtained from SHKM (adsorbent) waste*



In addition to adding various electrolytes and combustible materials to the catalyst composition, it is also possible to modify the catalyst properties by incorporating oxides of various metals (as active components). Various sources report that the oxides of *d*-metals and alkaline earth metals have a positive effect on catalyst properties. These metal oxides can be added to the catalyst by different methods and at different stages of the preparation process.

Calcium and magnesium oxides can be added in dry form, while their hydroxides can be introduced in suspension form (during the granulation process) as binder solutions. Calcium compounds, in particular, are considered relatively inexpensive.

To study the effect of CaO (as a promoter) on the catalyst properties, five different samples were prepared with varying compositions, and their physical and mechanical properties were analyzed.

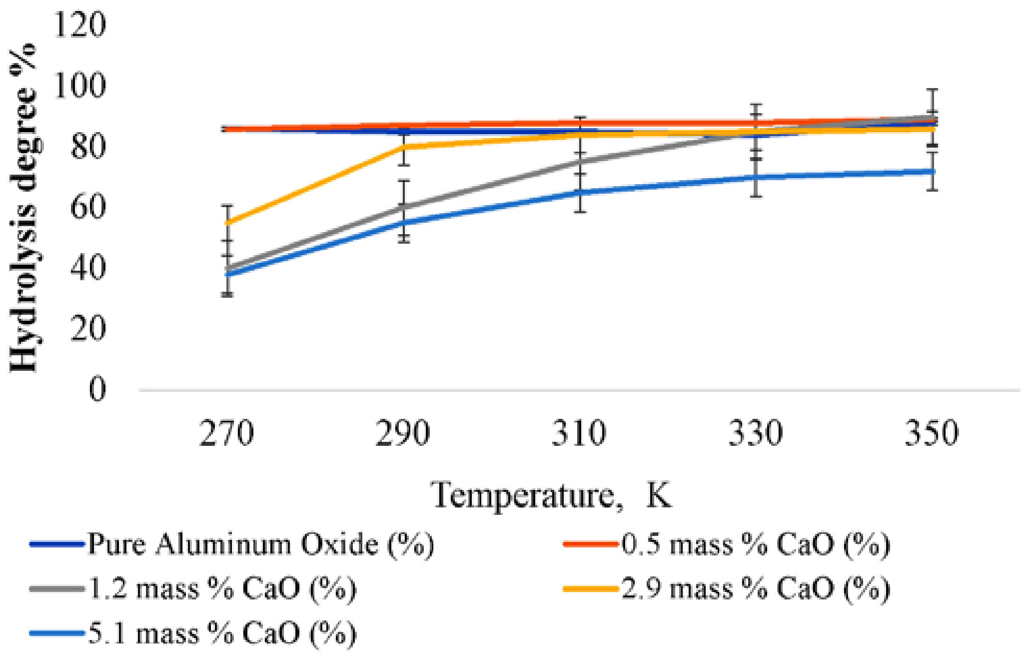
Table 4. *Main physical-chemical properties of catalysts obtained with different amounts of active component (CaO)*

Cao content (%)	Mechanical strength (mpa)	Total pore volume (cm^3/g)	Specific surface area (m^2/g)	Density (g/cm^3)
0	6.4	0.50	260	0.79
0.5	6.1	0.48	265	0.74
1.1	5.3	0.50	285	0.70
3.1	4.5	0.49	270	0.64
4.8	3.9	0.51	265	0.62

Analysis results showed that as the CaO content increased, the specific surface area increased while the density decreased (Table 4). It was also observed that with increasing CaO content, the mechanical strength of the granules decreased.

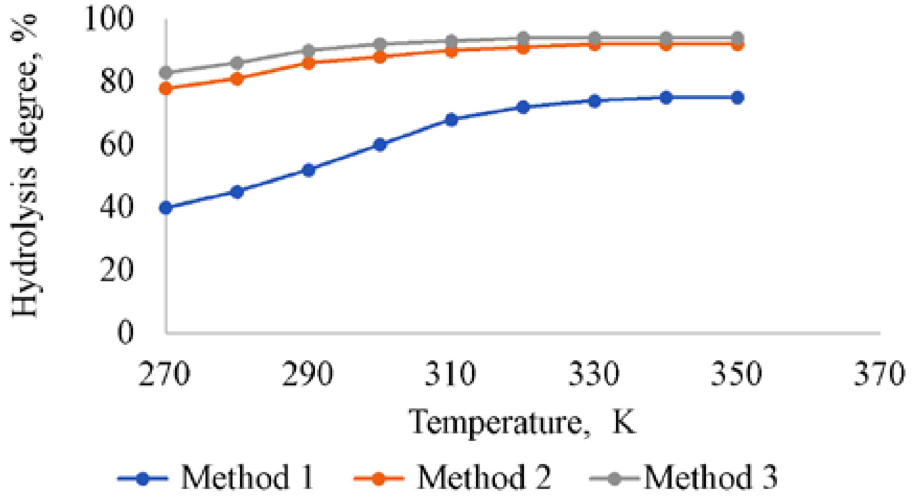
The temperature dependence of the carbon disulfide (CS_2) hydrolysis in the obtained catalyst samples was studied. The results showed that when the CaO content is 1%, the CS_2 hydrolysis reaction becomes temperature-independent. In contrast, when the CaO content is less than 1%, the reaction efficiency is dependent on temperature and decreases with temperature reduction.

Figure 3. *Temperature dependence of the hydrolysis degree of the reaction carried out on clean and actively component-loaded catalyst samples*



It was determined that the properties of the catalyst depend not only on its composition but also on its preparation method. it is evident that samples prepared by adding a $Ca(OH)_2$ suspension have a higher surface area and a lower density compared to samples prepared by other methods. The temperature dependence of the hydrolysis degree of carbon disulfide was studied on catalyst samples prepared by different methods. The results showed that the samples prepared by adding a $Ca(OH)_2$ suspension exhibited a higher hydrolysis degree. This can be explained by the better mixing of raw materials when using the active component suspension.

Figure 4. *Temperature dependence of the hydrolysis degree of the reaction carried out on catalyst samples prepared by different methods*



The addition of CaO to the catalyst composition resulted in an improvement in its properties. Specifically, the addition of CaO increased the catalyst's specific surface area and reduced its density. The overall hydrolysis reaction of CS_2 also improved. Based on the above analysis, it was determined that the optimal amount of the active component is 1%, because when the promoter amount exceeds 1%, the mechanical strength of the granules decreases, and other properties do not improve. Additionally, it was concluded that adding the active component to the catalyst in the form of a $Ca(OH)_2$ suspension is the most effective method.

Since a decrease in strength was observed when the CaO content exceeded 1%, it is considered appropriate to further study ways to enhance the strength of zeolite granules, especially under conditions with high amounts of active components, and to explore various metal oxides and their optimal quantities.

Conclusion

This study demonstrated that 10% NaOH increased microporosity from 0.05 to 0.18 cm^3/g and macroporosity from 0.26 to 0.31 cm^3/g; 8% cellulose reduced density from 0.712 g/cm^3 to 0.676 g/cm^3 and mechanical strength from 5.7 MPa to 4.2 MPa. An increase in CaO content from 0 to 4.8% enhanced specific surface area from 260 m^2/g to 265 m^2/g while decreasing density from 0.79 g/cm^3 to 0.62 g/cm^3. Adding $Ca(OH)_2$ suspension raised surface area to 295 m^2/g and density to 0.69 g/cm^3. The optimal composition was identified in catalysts containing 1% CaO (pore volume 0.37 cm^3/g) and 1% MgO (pore volume 0.41 cm^3/g). Thermal analysis confirmed boehmite formation at 250–300 °C and γ-Al_2O_3 formation at 500–700 °C.

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Contact: sciuzb@mail.ru