

CHARACTERIZATION OF SYNTHESIZED SILICA GEL FOR MOISTURE ADSORPTION BASED ON DTA-TGA AND BET ANALYSIS

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ANNOTATION.

This study presents the synthesis of silica gel under laboratory conditions using the sol–gel method based on sodium silicate (Na_2SiO_3) and sulfuric acid (H_2SO_4), along with a comprehensive investigation of its physicochemical properties. The synthesis process included gel formation, aging, washing, and controlled drying under different regimes to evaluate their influence on the material characteristics.

Thermal behavior and stability of the obtained samples were analyzed using DTA–TGA methods. The results revealed that mass loss in the temperature range of 40–150 °C corresponds to the removal of physically adsorbed water. Sample 2.2 Y demonstrated a higher moisture adsorption capacity (14.49%) and more developed porous structure, whereas sample 2.1 Y exhibited superior thermal stability due to the presence of structurally bound hydroxyl groups.

BET analysis showed that the synthesized silica gel possesses a specific surface area of 203.7 m^2/g and a total pore volume of 0.21 cm^3/g . The pore size distribution is predominantly within the range of 1.7–2.0 nm, indicating a mesoporous structure. Additionally, the presence of micropores was confirmed by t-method and HK analysis, contributing to enhanced adsorption properties.

The obtained results demonstrate that the synthesized silica gel has a well-developed porous structure and can be effectively applied as an adsorbent and desiccant material in various industrial processes.

KEYWORDS:

Silica gel; Sol-gel synthesis; BET analysis; Mesoporous structure; Adsorption; DTA-TGA; Porous materials; Desiccant

Kirish.

Silica gel is a highly porous amorphous silicon dioxide obtained by the sol-gel method, which absorbs moisture through physical adsorption. It is a stable, renewable and effective desiccant material widely used for humidity control in various industries.[1]

Silica gel is divided into groups A, B and C. Type A is mainly used for drying gases and protecting products from moisture. Type B is used for adsorbing liquids, controlling humidity and as a catalyst carrier. Type C is widely used in industrial gas drying, oil purification, pharmaceutical processes and other technological processes [2].

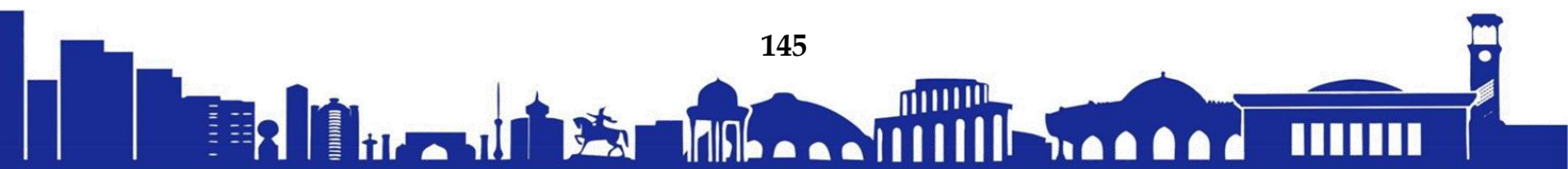
When silica gel types are modified with amines, their sorption capacity towards CO₂ increases. Silica gels modified with hydroxyethyl carbamate significantly increase CO₂ sorption due to the presence of amino acids. Optimal modification at 30% HEC doubles the sorption capacity, demonstrating high efficiency and stability, which makes these sorbents promising for CO₂ capture [3].

The addition of nano silica significantly enhances the strength and durability of concrete by promoting pozzolanic reactions and increasing C-S-H gel formation. Optimal nano silica content improves mechanical performance while reducing cement consumption, demonstrating its strong potential for advanced concrete applications [4].

Sol-gel methods are important in the preparation of hybrid and mesoporous materials such as SBA-15. Such materials are promising for adsorption and environmental applications due to their high surface area and functionalization potential. However, the development of simple and inexpensive laboratory protocols for their synthesis is an urgent challenge [5].

A microfluidic HPLC-mimicking system using silica gel-C18 was successfully applied for cyclotide separation. The method enables rapid, low-cost, and efficient separation of cyclotides, demonstrating strong potential for drug discovery applications [6].

Silica gel (SiO₂) is widely used as a sorbent, desiccant, and catalyst support. In Russia, the demand for it is high, and domestic production is insufficient, so it is imported in large quantities. Therefore, it is important to develop high-quality silica gel production based on local raw materials [7].



Materials.

Liquid glass (Na_2SiO_3), Sulfuric acid (H_2SO_4)

Methods.

DTA (Differential Thermal Analysis) — to determine the processes of heat absorption or release when a substance is heated.

TGA (Thermogravimetric Analysis) — to see how the mass of a substance changes with temperature.

BET (Brunauer–Emmett–Teller) method — for calculating the total surface area of a material by adsorption of a gas (usually nitrogen, N_2).

Experimental part.

In laboratory conditions, gelation was observed as a result of the interaction of liquid glass and sulfuric acid, and the resulting gel was washed and dried after a certain time.

In this case, sodium silicate and water were dissolved in a ratio of 1:4, 1:6, and the system was constantly stirred. Then, a diluted sulfuric acid solution was slowly added to it dropwise until a weakly acidic environment was formed. After the gel was formed, the stirring was stopped and the gel was allowed to rest for 12-24 hours. After the time was up, the gel was washed several times to remove the sodium sulfate salt formed in the system and filtered. The resulting gel was dehydrated at a temperature of 180-200 °C for 2 hours.

Drying process.

The samples were designated as 2.1 Y and 2.2 Y. During the drying process, both samples were dried in different ways. The temperature of both samples was increased to 180-200 °C for drying, and sample 2.1 Y was held at this temperature for 2 hours and then slowly cooled. Sample 2.2 Y was held at this temperature for 2 hours at 180-200 °C and then suddenly cooled. and the obtained samples were sent for testing.

When we examined the obtained samples through DTA–TGA analysis, the following results were obtained.

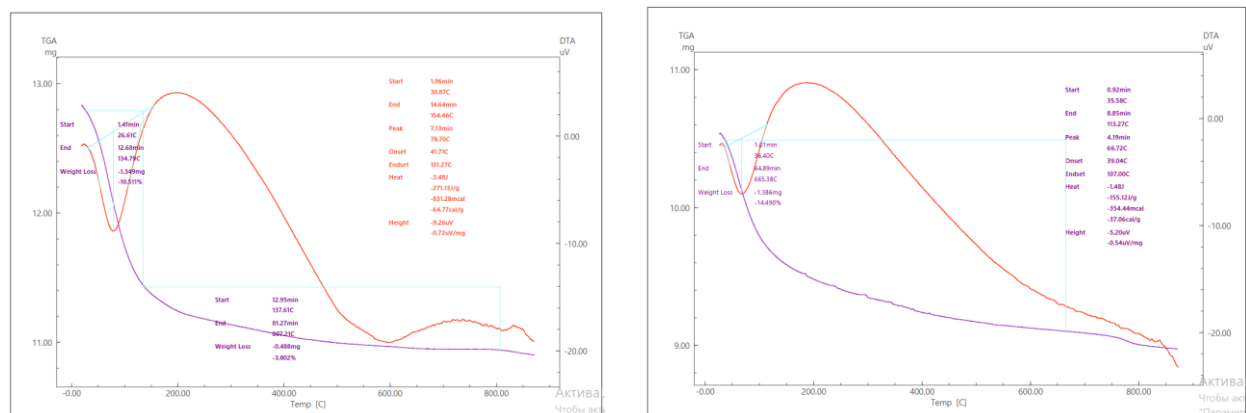


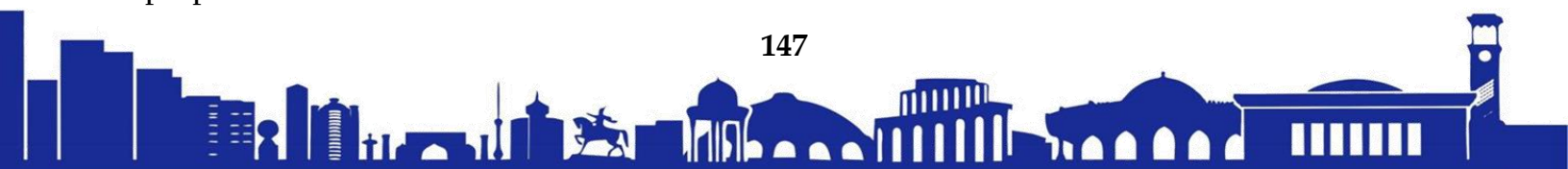
Figure 1. TGA–DTA results for the silica gel sample (2.1 Y and 2.2 Y).

TGA–DTA analysis of the synthesized silica gel samples showed that the mass loss in the range of 40–150 °C was due to the release of adsorbed water. Sample 2.2 Y had a high moisture absorption capacity (14.49%) and exhibited a developed porous structure. In sample 2.1 Y, the decomposition of structural hydroxyl groups was observed in an additional step, which revealed its high thermal stability. The results obtained confirm the high efficiency of silica gels as adsorbents.

Table 1. Comparative analysis of samples

Parameter	2.2 Y	2.1 Y
Moisture loss	High (14.49%)	Moderate (10.5%)
Peak temperature	66.7 °C	78.7 °C
Structural stability	Lower	Higher
Porosity	Very good	Good
Adsorption capacity	Very high	High

The BET analysis results of the synthesized silica gel sample are as follows. According to the results of BET analysis, the synthesized silica gel sample has a specific surface area of 203.7 m²/g, which indicates its well-developed pore structure. The total pore volume is 0.21 sm³/g. The pore sizes are mainly in the range of 1.7–2.0 nm, indicating that the material has a mesoporous structure. According to the results of the t-method and HK methods, the sample also contains micropores, which further enhances its adsorption properties.



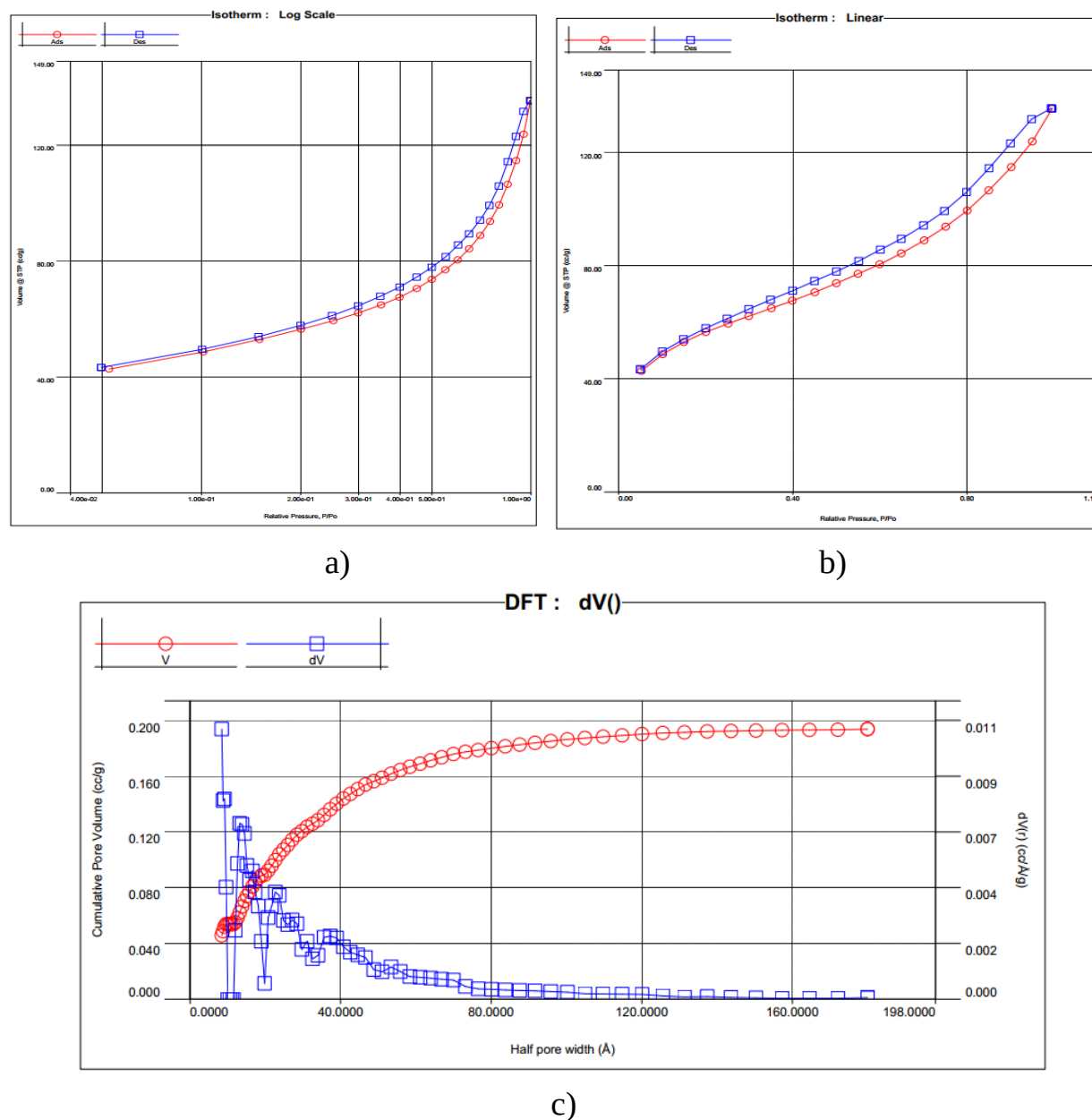
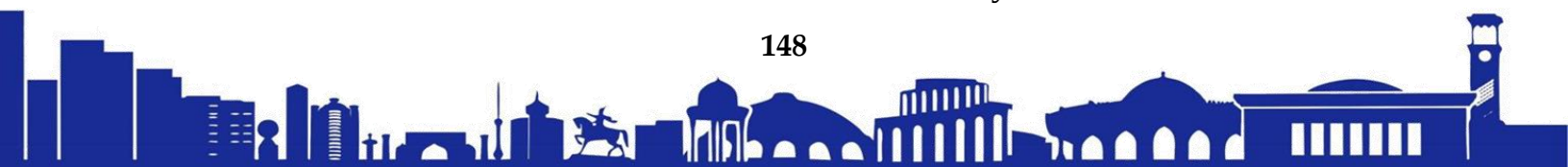


Figure 2. a), b) and c) - BET analysis results of the synthesized silica gel.

BET and pore structure analysis were performed according to the classical BET theory and IUPAC recommendations [8–10], and interpreted using modern approaches for porous materials [11–13].

CONCLUSION.

In this study, silica gel was successfully synthesized via a sol–gel method using sodium silicate and sulfuric acid under controlled laboratory conditions. The influence of



drying regimes on the structural and adsorption properties of the material was clearly demonstrated.

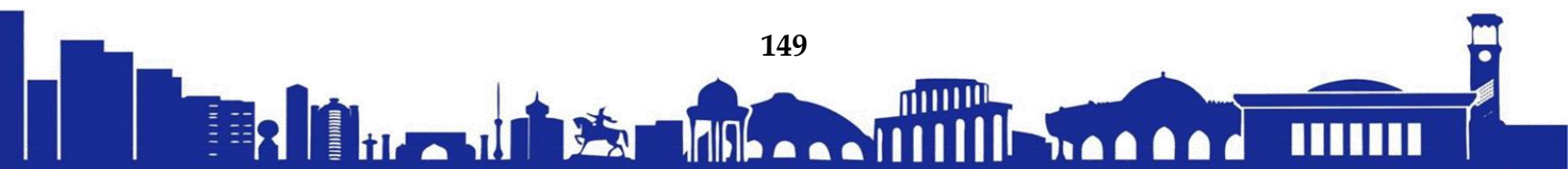
Thermal analysis confirmed that the synthesized samples exhibit typical dehydration behavior, with physically adsorbed water removed at low temperatures. The comparison of samples showed that rapid cooling enhances porosity and adsorption capacity, while slow cooling improves thermal stability.

BET analysis indicated that the silica gel possesses a relatively high specific surface area ($203.7 \text{ m}^2/\text{g}$) and a well-developed pore structure with both mesopores and micropores. The dominant pore size in the range of 1.7–2.0 nm confirms the mesoporous nature of the material, which is favorable for adsorption applications.

Overall, the synthesized silica gel demonstrates promising characteristics for use as an effective adsorbent in gas drying, moisture control, and environmental applications. Further modification (e.g., amine functionalization) could significantly enhance its performance for selective gas adsorption, particularly CO_2 capture.

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