

Synthesis and Physicochemical Characterization of Mesoporous Silica Gel Prepared by the Sol–gel Method

S. A. Tursunov

sarvarbektursunov26@gmail.com

Termez Institute of Engineering and Technology

S. Z. Xodjamkulov

Termez Institute of Engineering and Technology

M. U. Karimov

Tashkent Research Institute of Chemical Technology

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Abstract

In this study, silica gel was synthesized using the sol-gel method based on sodium silicate and sulfuric acid, and its physicochemical properties were comprehensively analyzed. The synthesized samples were characterized using DTA–TGA, BET, DFT, X-ray fluorescence (XRF) and FTIR methods. According to the results of TGA, it was found that silica gel has a mass loss of 14.49% and high thermal stability at temperatures above 600°C. As a result of BET analysis, the specific surface area was 203.7 m²/g, and according to the DFT method, it was 146.56 m²/g, the total pore volume was 0.194 cm³/g, and the material had a micro-mesoporous structure. XRF analysis showed that the silica gel contained 96.7 wt.% SiO₂, confirming the high chemical purity of the product. The detection of absorption bands characteristic of Si–O–Si and Si–OH functional groups in the FTIR spectrum indicated the successful formation of an amorphous silica skeleton. The results obtained indicate that the synthesized silica gel has high adsorption properties and can be used as a promising adsorbent for the effective adsorption of acidic gases by modification.

1.Introduction

Today, silica gel is widely used as a strategic material in many fields, such as adsorption, catalysis, chromatography, pharmaceuticals, food packaging, and gas drying, due to its high specific surface area, developed pore structure, and chemical inertness. In particular, mesoporous silica gels are gaining importance as catalyst carriers, controlled drug delivery systems, and promising adsorbents for CO₂ capture. The rapid development of the chemical, oil, gas, and food industries in Uzbekistan is steadily increasing the demand for high-quality adsorbents. The fact that a significant part of the country's need for silica gel is currently covered by imports makes the development of modern synthesis technologies based on local raw materials an urgent task. In this regard, the synthesis of mesoporous silica gel, which has the ability to control the structure and pore parameters using the sol-gel method, and the in-depth study of its physicochemical properties are scientifically and practically important, creating a solid scientific basis for the creation of local adsorbents with high added value, which can replace imports.

Today, the production of silica aerogels using the sol-gel method using atmospheric purification processing (APD) has been established as an effective method for obtaining materials with high permeability. The stages of gelation, aging, solvent injection and surface modification with Trimethylchlorosilane (TMCS) are of great importance in the formation of high-quality aerogels. In all, modification with TMCS converts the sliding process into a hydrophobic modification, ensuring the shrinkage of the material during processing and the preservation of the porous structure. The additional charge is considered a promising method for obtaining silica-based adsorbents with a high specific surface area and good adsorption properties [1].

In addition, it is possible to synthesize organosilicon compounds and obtain them using the traditional high-temperature carbothermic reaction method. The disadvantage of this method is that the process is accompanied by high energy consumption and the release of CO and CO₂ gases. Therefore, scientists

have proposed a new approach based on the conversion of quartz into amorphous silica gel ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$) using NH_4F . According to the results of the study, the transformation of crystalline quartz into amorphous silica gel increases the reactivity of the material and allows obtaining a product of high purity (more than 97.5% SiO_2). This approach is characterized by energy efficiency, environmental safety, and convenience for subsequent functional modification [2].

It has been reported in the literature that silica gel synthesized based on tetraethylorthosilicate (TEOS) by the sol-gel method has high adsorption properties. FTIR and XRD analyses confirmed the amorphous structure of silica gel, and adsorption experiments showed that it adsorbs methylene blue dye from aqueous solutions with high efficiency. Kinetic analyses revealed that the adsorption process conforms to a pseudo-second-order model. The results obtained indicate that silica gel is a promising material as a highly efficient and environmentally friendly adsorbent for the separation of not only dyes, but also other pollutants[3].

At the same time, it was found that when silica gel was added to the composite adsorbents, their specific surface area and adsorption efficiency increased significantly. In particular, scientists showed that chitosan nanocomposites modified with silica gel showed an adsorption capacity of up to 113 mg/g for Pd^{2+} ions and maintained high stability after several regeneration cycles. These results confirm the possibility of using silica gel as an effective adsorbent for the separation and recovery of heavy metal ions due to its developed pore structure and large surface area.[4]

Scientists have emphasized that the formation of silica gel is one of the important factors in improving the microstructure and mechanical properties of materials. In particular, it has been studied that highly polymerized silica gel formed by carbonation of $\beta\text{-C}_2\text{S}$ forms a dense microstructure and has high strength. The results of physicochemical analysis showed that silica gel reduces porosity and improves the bonding properties of the material. These results indicate the prospects of silica gel as a functional material with high structural stability [5].

In addition, researchers have studied the possibility of synthesizing high-quality NaA Zeolite from man-made silica gel generated from aluminum fluoride production. The results of the study showed that by purifying silica gel and optimizing the hydrothermal synthesis processes, it is possible to obtain NaA Zeolite with high ion exchange and water vapor adsorption properties [6].

SA Zabolotnykh et al. studied the adsorption properties of La(III) and Er(III) ions on silica gel modified with monoethanolamine. The results of the study revealed that the pH value, temperature and ionic composition of the solution significantly affect the adsorption efficiency. Kinetic and isotherm analyses showed that the pseudo-second-order model and the Langmuir isotherm fit well in describing the adsorption process. The results obtained showed that silica gel modified with monoethanolamine is an excellent adsorbent for the separation and concentration of rare earth metal ions [7].

Scientists also studied the moisture absorption and desorption properties of gypsum-based composites with the addition of silica gel. The results of the study showed that the porosity, moisture absorption and desorption efficiency of the composites increased significantly with the increase in the amount and particle size of silica gel. In particular, the moisture absorption capacity of the samples with the addition of 40% silica gel was 3.22 times higher than that of pure gypsum, and the material exhibited high moisture control stability. The results obtained confirm that silica gel has great practical value as a functional additive that effectively controls moisture in building materials.[8]

Funda Ulusu and Ali Bilgic synthesized silica gel modified with APTMS and pillar[V]arene and comprehensively studied its structural and biological properties. FTIR, SEM and TGA analyses confirmed that the modification process was successful and new functional groups were formed on the surface of the silica gel. The obtained material exhibited high antibacterial, antioxidant and cytotoxic activities, which indicates that the modification of silica gel with various functional groups significantly expands its application possibilities [9].

In this research work, the properties of silica gel, its applications, and the synthesis of various silica gel-based materials and their properties were analyzed. In addition, the synthesis of silica gel by the sol-gel method using sodium silicate and sulfuric acid in laboratory conditions, the optimal process conditions were studied. The samples were also subjected to physicochemical analysis and the analysis of the results was presented.

2. Materials and methods

2.1. Materials.

Liquid glass (Na_2SiO_3) - purchased from the manufacturer OOO "TECH SNAB" via the website glotr.uz. Chemically pure sulfuric acid (H_2SO_4) (GOST 4204-77) - purchased from OOO "Chemical Invest". Sodium silicate (liquid glass) was used as the silica precursor for silica gel synthesis and sulfuric acid was used as a catalyst and pH-adjusting agent during the preparation process.

2.2. Methods

DTA, TGA analysis

Differential thermal analysis (DTA, Shimadzu DTG-60H) was used to study the heat absorption and heat release processes occurring during heating of the synthesized silica gel samples. Thermogravimetric analysis (TGA, Shimadzu DTG-60H) was performed to evaluate the thermal stability and mass loss properties of the silica gel as a function of temperature.

BET analysis

The specific surface area of the silica gel was determined by the BET method using nitrogen adsorption analysis on the AsiQwin™ Version 5.21 instrument (software) manufactured by Qutachrome Instruments.

X-ray fluorescence analysis

The chemical composition of the synthesized silica gel was determined using a Rigaku NEX CG EDXRF energy dispersive X-ray fluorescence spectrometer.

FTIR analysis

Fourier transform infrared spectroscopy (IN-Fi spectrometer, Shimadzu, Japan) was used to determine the function spectrum and bond structure of the silica gel.

2.3. Experimental part.

We used a solution of sodium orthosilicate (liquid glass), which is commercially available as a building material, for the synthesis of silica gel. The reaction process was carried out based on the Sol-gel method determined as a result of literature analysis. The resulting liquid glass was diluted with distilled water in a mass ratio of 1:4 to prepare a solution of 100 g in the range of 10–20% and mixed thoroughly. In another glass vessel, 100 ml of a 15% solution of sulfuric acid was prepared. The sodium orthosilicate solution was stirred continuously and sulfuric acid solution was added dropwise to it until a gel was formed. When the solution medium (pH 3–5) became weakly acidic, the acid was stopped dropping. The mixture was stirred for 12 hours until a complete gel was formed and washed 3 times with distilled water to remove the formed sodium sulfate salt. The solution was filtered and then heated at 180–200 °C for 2 hours to remove the water contained in the gel. After the time was up, the heated gel was suddenly cooled. In this case, the gel retained its amorphous state. The resulting gel was transparent and brittle, which is a silica gel material obtained in laboratory conditions. The porosity of the resulting material directly depends on the pH level at the point of completion of the neutralization process of the solution and the gelation temperature of the solution.

The reaction for the formation of silica gel is written as follows:

Figure 1 illustrates the process of silica gel formation by the neutralization reaction of sodium silicate with sulfuric acid. During the reaction, hydrogen ions from sulfuric acid displace sodium ions from sodium silicate, forming a hydrated silica gel ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$). The resulting gel is washed to remove soluble byproducts such as sodium sulfate, and a highly porous silica gel is obtained. This method is simple, cost-effective, and provides a high-purity adsorbent for silica gel synthesis.

3. Results and Its Discussion

3.1. FTIR analysis

In addition, IR spectroscopy (FTIR) analysis was performed to determine the molecular structure and functional groups of the substance, and the following results were obtained.

FTIR spectroscopy was used to determine the chemical structure of the silica gel. The results of the analysis confirmed that the synthesized silica gel had an amorphous silica structure. The broad absorption band observed in the spectrum at 3334.92 cm^{-1} corresponds to the O–H stretching vibrations of silanol (Si–OH) groups and adsorbed water molecules. The peak at 1647.21 cm^{-1} indicates the $\delta(\text{H–O–H})$ deformation vibration of adsorbed water. The strong absorption band observed at 1062.78 cm^{-1} represents the asymmetric stretching vibration of Si–O–Si bonds inherent in the silica gel framework. The peaks at 825.53 and 790.81 cm^{-1} indicate the symmetric vibrations of Si–O–Si bonds and correspond to Si–O bonds. The absorption at 493.78 cm^{-1} is associated with the deformation vibration of Si–O–Si bonds. The obtained results indicate that the synthesized sample contains characteristic functional groups of silica gel and a highly structured silicon oxide skeleton.

3.2. TGA–DTA analysis of synthesized silica gel

According to the results of differential thermal analysis, a heat absorption peak was observed at 66.72°C in the DTA curve. This indicates the presence of moisture, i.e. physically adsorbed water, in the capillary pores of the sample. This is typical for silica gel.

Table 3.1

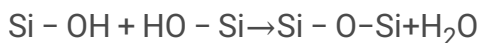
Thermal stability and mass change upon heating of the synthesized silica gel sample based on TGA–DTA results.

Parameter	Value
Onset temperature	39.04°C
Endset temperature	107.00°C
DTA peak temperature	66.72°C
Total weight loss	1.386 mg
Weight loss	14.49%
Heat effect (Enthalpy)	-155.12 J/g
Final TGA temperature (weight-loss range)	665.38°C

After 100°C , no new large endothermic or exothermic peaks were observed in the DTA curve. This indicates that the synthesized silica gel sample does not contain organic matter and no additional decomposition has occurred. It can be concluded that the synthesized sample is highly pure.

The results of thermogravimetric analysis showed that a mass loss of 14.49% was observed. This phenomenon indicates that the pore structure of the obtained silica gel is well developed and water molecules are effectively retained in these pores. A mass loss in the range of 10–20% is usually considered a normal value for silica gels.

In the range of 600–800°C, the TGA curve gradually decreases. This is due to the condensation of silanol (Si–OH) groups, the elimination of water, and the formation of Si–O–Si bonds.



As can be seen from the graph, after 100°C the mass remains almost stable and no sharp decomposition is observed up to 600–700°C. This indicates that silica gel has high thermal stability.

3.3. BET ANALYSIS

3.3.1. BJH analysis

The pore size distribution analysis performed using the BJH (Barrett–Joyner–Halenda) method revealed that the synthesized silica gel sample had well-developed mesoporous pores, and the total pore volume of the sample was 0.136 sm³/g and the total surface area was 71.435 m²/g. Accordingly, the calculated average pore radius was 17.073 Å and the pore diameter was approximately 3.41 nm. This value was found to fall within the range of mesoporous (2–50 nm) according to the IUPAC classification.

Figure 3.3. Classification of pores by size.

According to the BJH desorption curve, the bulk of the pore volume is concentrated in the radius range of 17–60 Å. In particular, at a radius of 17.07 Å, the differential pore volume ($dV(r) = 0.00413 \text{ sm}^3/\text{Å/g}$) and differential surface area ($dS(r) = 4.84 \text{ m}^2/\text{Å/g}$) are close to their maximum values, indicating that the majority of the pores in the sample correspond to this size. This indicates that the gradual decrease in the values of $dV(r)$ and $dS(r)$ with increasing radius indicates that the pores are relatively uniformly distributed and the proportion of large macropores is low. The cumulative pore volume was observed to increase steadily with increasing radius, reaching a value of 0.136 sm³/g. This indicates that there are sufficient mesopores within the adsorbent pores for free diffusion of gas molecules. The cumulative surface area is 71.435 m²/g, which confirms the sufficient development of active centers during the adsorption process.

3.3.2. MBET (Brunauer–Emmett–Teller) analysis.

According to the results of MBET (Brunauer–Emmett–Teller) analysis, 4 points were selected on the surface of the silica gel and the specific surface area, adsorption processes and pore formation were determined using nitrogen (N₂) adsorbate at 77.35 K (liquid nitrogen purification). According to this, a silica gel sample of 0.1138 g was taken and it took 3.3 hours at 120 °C to remove moisture and volatile power. BET const. 123.893, correlation coefficient 0.999930, and the total internal surface area of the obtained silica gel sample was 203.7 m²/gr.

Table 3.2
Comparative analysis of silica gel surfaces

Material	BET Surface Area (m ² /g)
Conventional silica gel	150–300
Laboratory-synthesized high-quality silica gel	200–500
Synthesized silica gel	146,56 -203.68

As can be seen from the comparison table above, the obtained value of 203.68 m²/g indicates that the silica gel was successfully synthesized.

The specific surface area of the silica gel sample was determined using the multipoint BET method. Experimental points in the range of relative pressure 0.052–0.200 were selected for calculation, since this range corresponds to monolayer adsorption conditions. With increasing relative pressure, the adsorbed nitrogen volume increased steadily from 42.77 sm³/g to 56.50 sm³/g. The good fit of the obtained points to the linear form of the BET equation confirms that the adsorption process proceeded in accordance with the requirements of the model and the reliability of the experimental results. The observed adsorption properties indicate that the silica gel has a developed porous structure. The presence of micro- and small mesopores, determined by DFT analysis, supports this conclusion. Thus, the large internal surface area of the material and the large number of active centers allow it to be evaluated as a promising adsorbent for effective CO₂ capture.

Table 3.3
Multi-Point BET Data

Relative Pressure (P/Po)	Volume (c.c./g)	1 / [W((Po/P) - 1)] (1/g)
0.0524288	42.7710	1.0350
0.101320	48.6798	1.8531
0.149925	52.9678	2.6641
0.200349	56.5023	3.5479

3.3.3. DFT analysis

According to DFT analysis, the synthesized silica gel sample has a moderately developed porous structure. The specific surface area is 146.56 m²/g, and the total pore volume is 0.194 sm³/g. The most probable half-width of the pores is 8.44 Å (full width is about 1.7 nm), which indicates that the sample has a predominantly microporous–small mesoporous structure. The very small fitting error (0.365%) confirms the reliability of the calculation results. In general, although this silica gel has high dispersion, the surface area is average compared to classical high-surface silica gels.

Table 3.4
Conclusion on the DFT method

Parameter	Value
Pore volume	0.194 sm^3/g
Specific surface area	146.560 m^2/g
Lower confidence limit	8.440 Å
Fitting error	0.365%
Pore half-width (mode value)	8.440 Å
Moving-point averaging	Disabled

Analysis of the pore size distribution obtained using the DFT (Density Functional Theory) method. According to the results of the DFT analysis, it was found that the pore structure of the synthesized silicagel consists mainly of micro and small mesopores. The highest intensity in the differential pore size distribution (dV) curve is observed in the range of approximately 8–12 Å, indicating that pores of this size predominate in the sample. In addition, there is a second, relatively small peak in the range of 15–25 Å, indicating that small mesopores are also formed. At sizes above 30 Å, the differential pore size decreases sharply, and after 60 Å it becomes almost zero. This indicates that the amount of large mesopores and macropores is very small.

The cumulative pore volume (V) curve shows that the bulk of the pore volume is formed in the range of 5–50 Å. The curve increases sharply in this range and remains almost unchanged after 80 Å. The final total pore volume is approximately 0.20 sm^3/g . This result confirms that the bulk of the pores of the silica gel are small in size and the proportion of large pores is very low.

According to the IUPAC classification, pores up to 20 Å (2 nm) are micropores, and pores in the range of 20–500 Å (2–50 nm) are mesopores. Based on the results of DFT analysis, the studied silica gel can be characterized as a micro–mesoporous adsorbent. Such a pore structure is favorable for the adsorption of gas molecules, especially CO_2 .

Since the kinetic diameter of the CO_2 molecule is approximately 3.3 Å, pores in the range of 10–20 Å are considered the optimal size for the adsorption of CO_2 molecules. Therefore, the obtained DFT results indicate that the synthesized silica gel has the ability to effectively adsorb CO_2 . However, the total pore volume of about 0.20 sm^3/g is slightly lower than that of industrial silica gels, indicating that the adsorption capacity can be further increased by modifying it with amine compounds or other functional modifiers in the future. In general, the results of DFT analysis confirm that the synthesized silica gel has a well-developed micro-mesoporous structure, the majority of the pores are located in the range of 10–30 Å, and this structure is favorable for the selective adsorption of CO_2 gas. These properties allow the use of this silica gel as a promising adsorbent in gas separation and carbon dioxide capture processes.

Table 3.5
Hole size distribution by DFT method

Half pore width (Å)	Pore Volume (n.sh./g)	Surface Area (m ² /g)	dV(r) (n.sh./Å/g)	dS(r) (m ² /Å/g)
8.4397	0.045783	87.193	0.010500	12.4410
8.8282	0.048782	90.590	0.0077203	8.7451
9.2345	0.051944	94.014	0.0077815	8.4266
9.6595	0.053794	95.930	0.0043532	4.5067
10.1041	0.053794	95.930	0.0000000	0.0000
10.5692	0.053794	95.930	0.0000000	0.0000
11.0557	0.053794	95.930	0.0000000	0.0000
11.5646	0.053794	95.930	0.0000000	0.0000
12.0969	0.055220	97.108	0.0026772	2.2131
12.6537	0.058157	99.429	0.0052757	4.1693
13.2361	0.062138	102.440	0.0068345	5.1636
13.8453	0.066275	105.430	0.0067918	4.9055
14.4826	0.070380	108.260	0.0064416	4.4478
15.1492	0.073845	110.550	0.0051973	3.4307
15.8465	0.077072	112.580	0.0046281	2.9206
16.5758	0.080708	114.780	0.0049854	3.0076
17.3388	0.083888	116.610	0.0041674	2.4035
18.1369	0.086770	118.200	0.0036114	1.9912
18.9717	0.088645	119.190	0.0022465	1.1842
19.8449	0.089180	119.460	0.00061219	0.30849
20.7583	0.092072	120.850	0.0031660	1.5252
21.7138	0.095531	122.440	0.0036207	1.6675
22.7132	0.099693	124.280	0.0041645	1.8335
23.7586	0.103900	126.050	0.0040267	1.6948
24.8522	0.107250	127.390	0.0030572	1.2302
25.9961	0.110560	128.670	0.0028960	1.1140

Half pore width (Å)	Pore Volume (n.sh./g)	Surface Area (m ² /g)	dV(r) (n.sh./ Å/g)	dS(r) (m ² / Å/g)
27.1926	0.114240	130.020	0.0030757	1.1311
28.4443	0.117910	131.310	0.0029327	1.0310
29.7535	0.120440	132.160	0.0019348	0.65030
31.1230	0.123520	133.150	0.0022496	0.72280
32.5555	0.125770	133.840	0.0015693	0.48202
34.0540	0.128320	134.590	0.0016993	0.49901
35.6214	0.132080	135.650	0.0024017	0.67423
37.2610	0.136110	136.730	0.0024550	0.65888
38.9760	0.140180	137.770	0.0023716	0.60848
40.7700	0.143820	138.670	0.0020330	0.49866
42.6465	0.147250	139.470	0.0018246	0.42783
44.6095	0.150630	140.230	0.0017235	0.38634
46.6628	0.153950	140.940	0.0016153	0.34616
48.8105	0.156410	141.440	0.0011463	0.23486
51.0572	0.158800	141.910	0.0010655	0.20869
53.4072	0.161740	142.460	0.0012515	0.23434
55.8655	0.164380	142.930	0.0010718	0.19186
58.4368	0.166630	143.320	0.00087608	0.14992
61.1265	0.168930	143.690	0.00085315	0.13957
63.9401	0.171220	144.050	0.00081444	0.12738
66.8831	0.173500	144.390	0.00077445	0.11579
69.9616	0.175760	144.720	0.00073572	0.10516
73.1818	0.177340	144.930	0.00049100	0.067092
76.5502	0.178690	145.110	0.00040105	0.052394
80.0736	0.180020	145.280	0.00037776	0.047174
83.7592	0.181340	145.430	0.00035798	0.042742
87.6145	0.182650	145.580	0.00033870	0.038657

Half pore width (Å)	Pore Volume (n.sh./g)	Surface Area (m ² /g)	dV(r) (n.sh./Å/g)	dS(r) (m ² /Å/g)
91.6472	0.183930	145.720	0.00031822	0.034720
95.8655	0.185190	145.850	0.00029744	0.031029
100.2780	0.186370	145.970	0.00026773	0.026700
104.8936	0.187330	146.060	0.00020918	0.019941
109.7216	0.188290	146.150	0.00019868	0.018106
114.7718	0.189250	146.230	0.00018976	0.016533
120.0545	0.190200	146.310	0.00017988	0.014985
125.5804	0.190910	146.370	0.00012873	0.010250
131.3606	0.191380	146.410	0.000081237	0.0061825
137.4068	0.191960	146.450	0.000096016	0.0069881
143.7314	0.192390	146.480	0.000067935	0.0047263
150.3470	0.192700	146.500	0.000047163	0.0031368
157.2672	0.192910	146.510	0.000029948	0.0019051
164.5059	0.193120	146.530	0.000028916	0.0017580
172.0777	0.193330	146.540	0.000027247	0.0015839
179.9981	0.193740	146.560	0.000052313	0.0029052

3.3.4. Isotherm: Linear and Log Scale

Nitrogen adsorption-desorption isotherms recorded at 77.35 K showed that the amount of adsorbed nitrogen gradually increased over the entire range of relative pressures, indicating the presence of a well-developed porous structure in the synthesized silica gel. When (P/P₀) was close to unity, the adsorption network reached approximately 135 sm³/g, while the desorption network exhibited a moderate hysteresis loop, typical of a material containing micro- and mesopores. The texture parameters obtained from the isotherm analysis showed a surface area of 146.56 m²/g and a total pore volume of 0.194 sm³/g. The pore half-width corresponding to the modal value was 8.44 Å, which confirms the contribution of narrow pores to the overall structure. The low fit error (0.365%) indicates the high reliability of the applied model. These results indicate that the synthesized silica gel has an enhanced internal surface area and a pore architecture favorable for the adsorption and selective capture of CO₂ molecules. Adsorption measurements were performed using nitrogen at 77.35 K on an Autosorb iQ analyzer after degassing the sample at 120 °C.

The adsorption–desorption isotherm of nitrogen at 77 K showed a continuous increase in the adsorbed gas volume with increasing relative pressure in the synthesized silicagel. While the intensive adsorption observed at low relative pressures indicates the presence of micropores, the gradual increase in the high pressure region confirms the contribution of mesoporous ones. At conditions close to $P/P_0 \approx 1$, the adsorbed nitrogen volume reached $135 \text{ cm}^3/\text{g}$, indicating that the material has a well-developed porous structure. The overall shape of the isotherm confirms that the silicagel has sufficient internal surface area and pore volume to effectively trap CO_2 molecules. These results are in good agreement with the surface area and pore parameters determined by BET and DFT analyses.

Table 3.6
Experimental data on the nitrogen adsorption–
desorption isotherm of the synthesized silicagel (77
K).

Nº	Relative Pressure (P/0P₀)	Volume (sm³/g)
1	0.0524288	42.7710
2	0.1013200	48.6798
3	0.1499250	52.9678
4	0.2003490	56.5023
5	0.2513780	59.4641
6	0.2988510	62.1107
7	0.3509460	64.9038
8	0.3987170	67.5284
9	0.4505280	70.5556
10	0.5000630	73.6966
11	0.5495620	77.0321
12	0.5994360	80.4212
13	0.6489220	84.2626
14	0.7014880	88.8527
15	0.7509200	93.6925
16	0.8004200	99.3954
17	0.8497900	106.5147
18	0.9013670	114.7187
19	0.9499520	123.7886
20	0.9952140	135.3773
Nº	Relative Pressure (P/P₀)	Volume (sm³/g)
21	0.9928690	135.4481
22	0.9481230	131.6642
23	0.8997850	123.0603
24	0.8503080	114.3259

Nº	Relative Pressure (P/0P ₀)	Volume (sm ³ /g)
25	0.7988910	105.8944
26	0.7479230	99.2194
27	0.7001650	94.1260
28	0.6487930	89.3967
29	0.6008620	85.5836
30	0.5512720	81.5536
31	0.5001760	77.8719
32	0.4496100	74.4875
33	0.3994750	71.0684
34	0.3488130	67.8865
35	0.2988600	64.5712
36	0.2488810	61.2164
37	0.1999000	57.8457
38	0.1490610	53.9540
39	0.1005130	49.5799
40	0.0496370	43.3030

3.4. X-ray fluorescence (XRF) analysis

When X-ray fluorescence (XRF) analysis was performed to identify the chemical elements in the sample and measure their amounts (percentage or ppm), the following results were obtained.

This graphic image shows the XRF spectrum of the synthesized silica gel sample. The characteristic peaks corresponding to the element Si have the highest intensity in the spectrum, confirming that silica is the main component in the sample. In addition, signals corresponding to the elements Na, Al, Fe, Ca, Ti and Zr are observed at low intensity, which is consistent with the results of the XRF quantitative analysis. The peaks corresponding to the remaining elements have very low intensity, indicating that they are trace impurities.

The oxide composition of the sample obtained based on the data of the XRF spectrum is presented in a table below.

Table 3.7
Chemical composition of the synthesized sample and their amounts
(percentage or ppm)

No.	Component	Result	Unit	Stat. Err.	LLD	LLQ
1	Cl	0.0073	mass%	0.0002	0.0002	0.0007
2	Na ₂ O	2.27	mass%	0.301	0.431	1.29
3	Al ₂ O ₃	0.567	mass%	0.0132	0.0326	0.0979
4	SiO ₂	96.7	mass%	0.0637	0.0494	0.148
5	SO ₃	0.0784	mass%	0.0013	0.0012	0.0036
6	K ₂ O	0.0261	mass%	0.0016	0.0026	0.0079
7	CaO	0.0760	mass%	0.0019	0.0016	0.0047
8	TiO ₂	0.0627	mass%	0.0011	0.0009	0.0028
9	V ₂ O ₅	(0.0014)	mass%	0.0004	0.0011	0.0033
10	Cr ₂ O ₃	0.0013	mass%	0.0001	0.0003	0.0010
11	MnO	0.0067	mass%	0.0004	0.0007	0.0021
12	Fe ₂ O ₃	0.118	mass%	0.0012	0.0006	0.0019
13	CuO	0.0015	mass%	< 0.0001	0.0002	0.0006
14	ZnO	0.0019	mass%	< 0.0001	0.0001	0.0003
15	SrO	0.0024	mass%	< 0.0001	< 0.0001	0.0001
16	Y ₂ O ₃	0.0010	mass%	< 0.0001	< 0.0001	0.0002
17	ZrO ₂	0.0896	mass%	0.0012	0.0003	0.0008
18	Ag ₂ O	0.0005	mass%	< 0.0001	< 0.0001	0.0003
19	Ta ₂ O ₅	0.0015	mass%	0.0002	0.0004	0.0012
20	PbO	0.0007	mass%	< 0.0001	0.0001	0.0003
21	Dy ₂ O ₃	0.0048	mass%	0.0006	0.0014	0.0043

According to the XRF analysis, the composition of the prepared silica gel is SiO₂, the amount of which is 96.7 mass.%. This result confirms that the silica gel of high purity was obtained. The composition of Na₂O (2.27 mass.%) and Al₂O₃ and (0.567 mass.%) is clearly increased, while Fe₂O₃ (0.118 mass.%),

ZrO₂ (0.0896 mass.%), SO₃ (0.0896 mass.%), SO₃ (0.0784%) (0.0784%). TiO₂ (0.0627 mass.%) was less noticeable. The remaining oxides were detected in trace amounts (< 0.03 mass.%) and did not affect the overall composition of the silica gel.

Typically, the mass fraction of (SiO₂) in silica gel is 95–99%. The results show that the mass fraction of (SiO₂) in the synthesized silica gel sample is 96.7%. This proves that the synthesized silica gel has high purity. The presence of sodium oxide (Na₂O) in its composition confirms that it is obtained on the basis of sodium silicate (Na₂SiO₃).

Conclusion

The following conclusions were drawn as a result of the research: The conditions for the successful synthesis of silicagel using the sol-gel method based on sodium silicate were studied. As a result of a comprehensive analysis of the physicochemical properties of the synthesized silicagel, the content of SiO₂ in the synthesized sample according to XRF indicators was 96.7 mass.%, confirming the high chemical purity of the product. The detection of absorption bands characteristic of Si–O–Si and Si–OH bonds in the FTIR spectrum indicated the successful formation of an amorphous silicagel structure.

As a result of BET and DFT analyses, it was determined that the silicagel has a developed porous structure. The specific surface area of the sample is 203.7 m²/g, the total pore volume is 0.194 sm³/g, which confirm its high adsorption properties. The adsorption–desorption isotherm showed features characteristic of a mesoporous structure. DTA–TGA analysis showed high thermal stability of the material up to 600°C.

The results confirm that the synthesized silicagel is an adsorbent with high purity, enhanced surface area, and thermal stability. These properties indicate that it is a promising material for further modification as an effective sorbent for the adsorption of CO₂ and other acidic gases.

Declarations

Author Contribution

Barcha mualliflar qo'lyozmani ko'rib chiqishgan

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Figures

Image not available with this version

Figure 1

Legend not included with this version

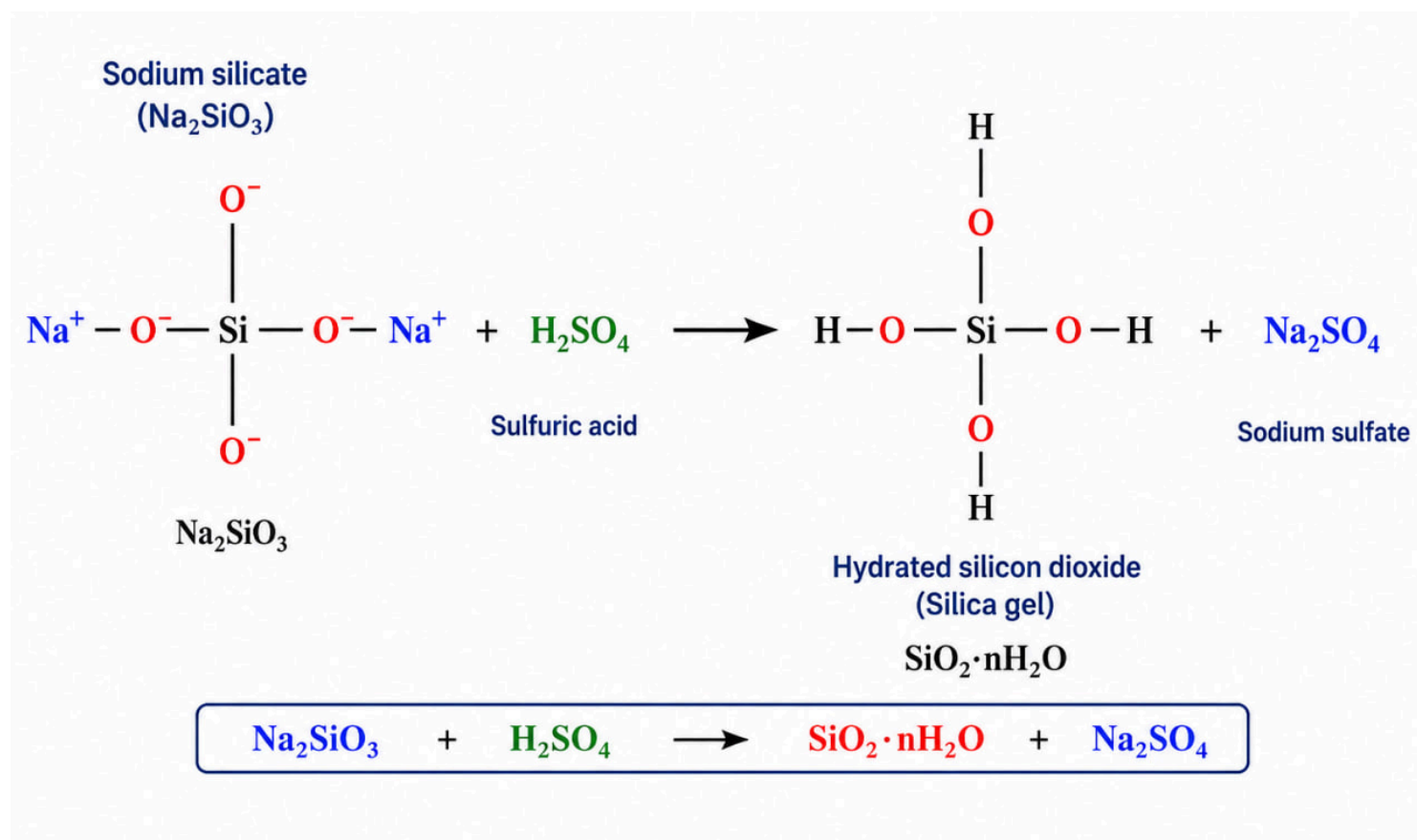


Figure 2

Figure 2.1. Reaction of sodium silicate with sulfuric acid

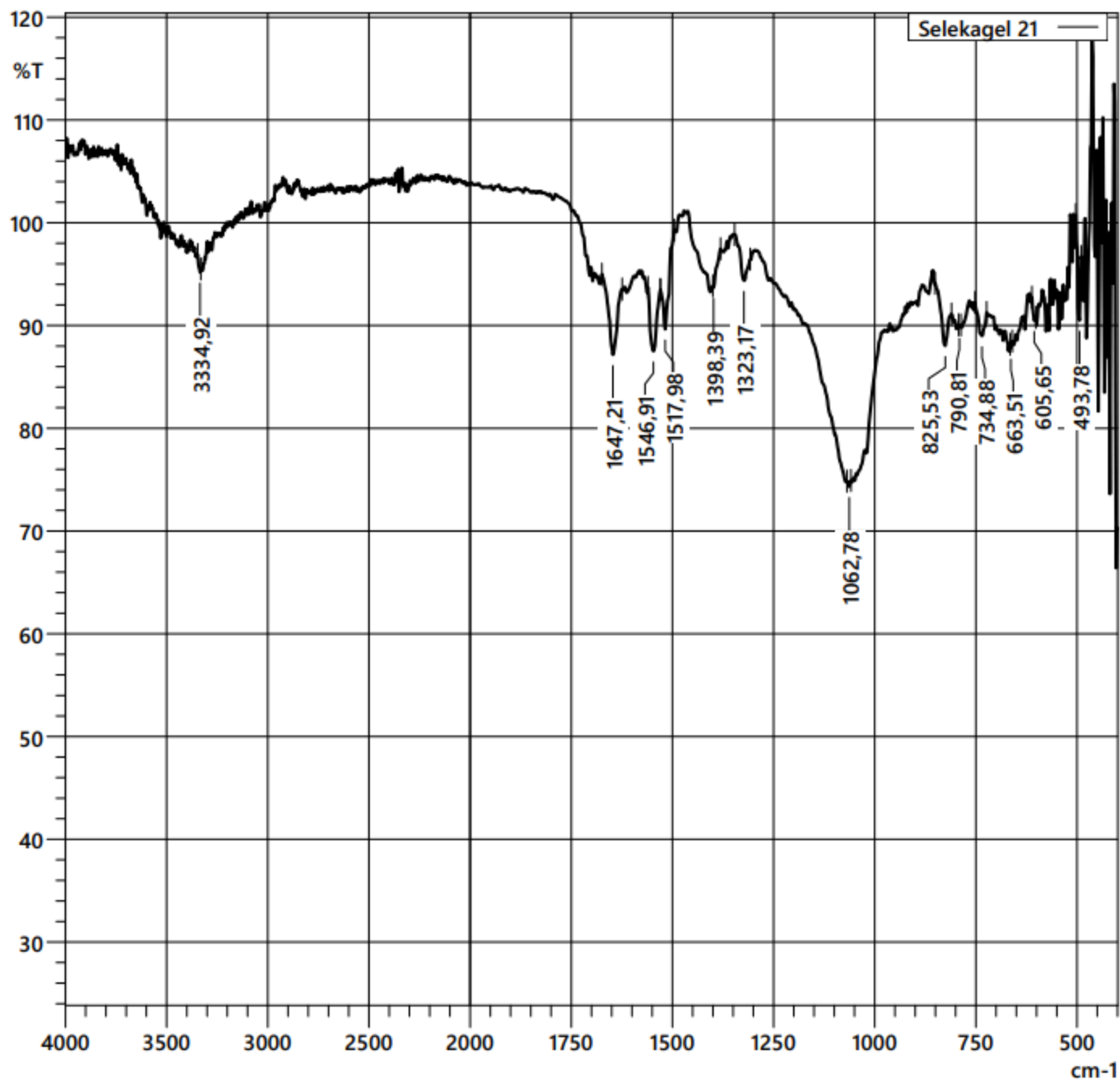


Figure 3

Figure 3.1. IR spectroscopy (FTIR) analysis of the synthesized silica gel sample.

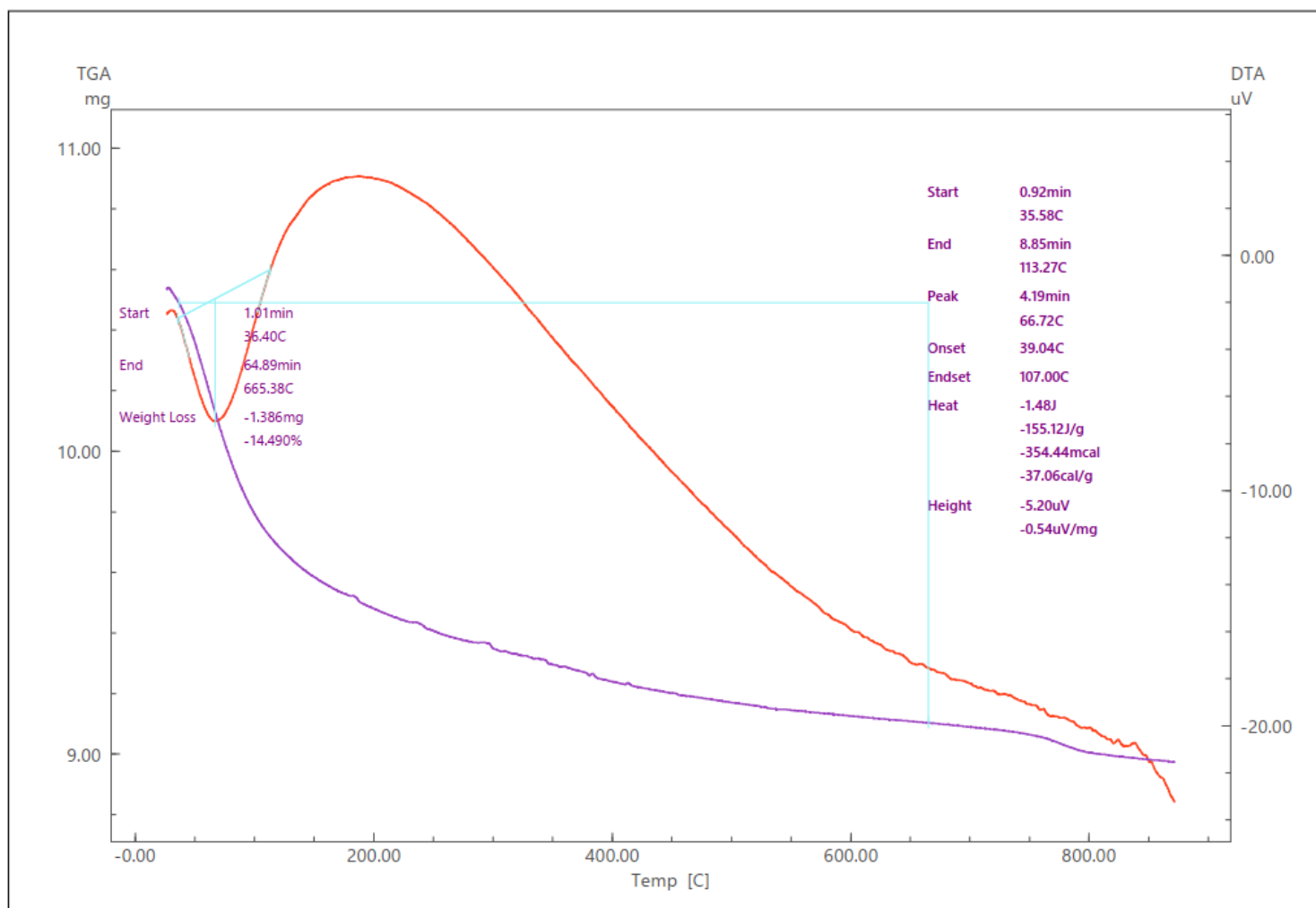


Figure 4

Figure 3.2. TGA–DTA results.



Figure 5

Figure 3.3. Classification of pores by size.

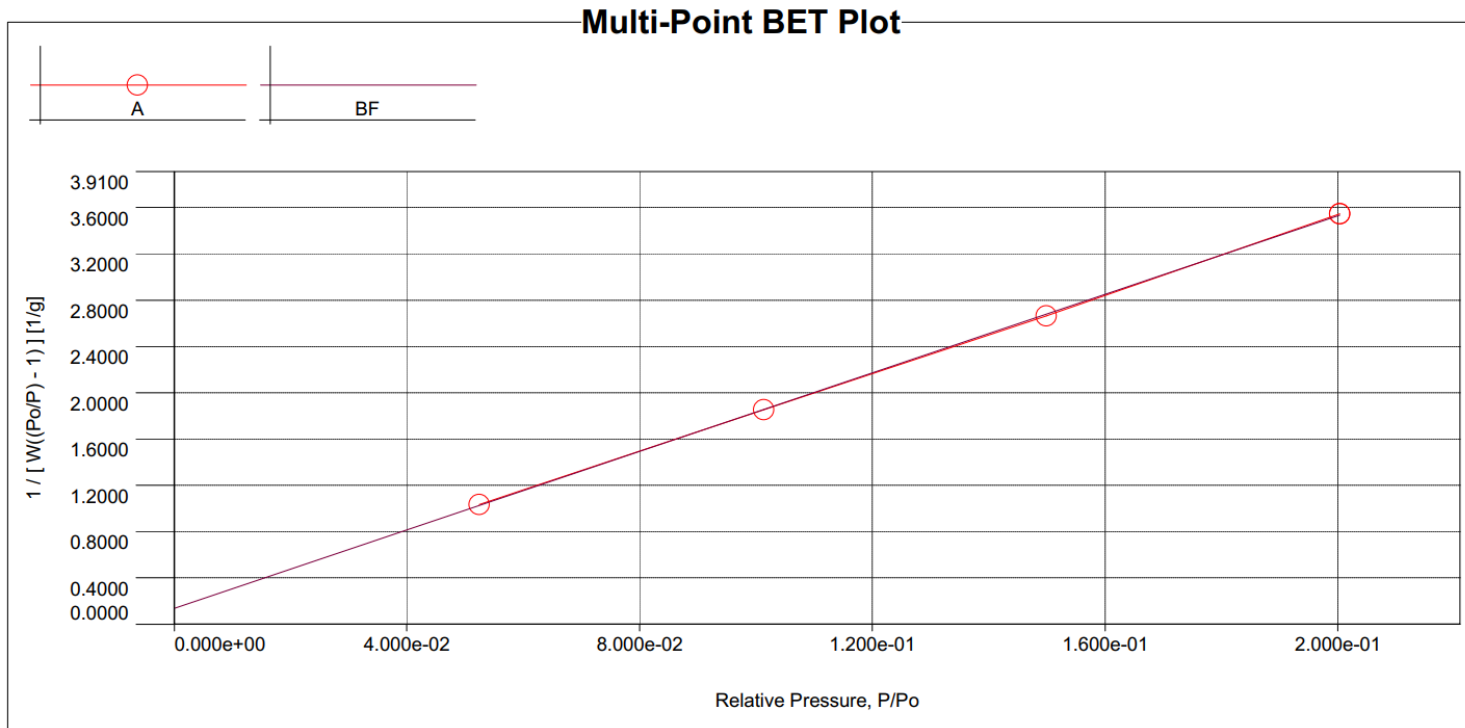


Figure 6

Figure 3.4. Multi-Point BET plot of the synthesized silica gel.

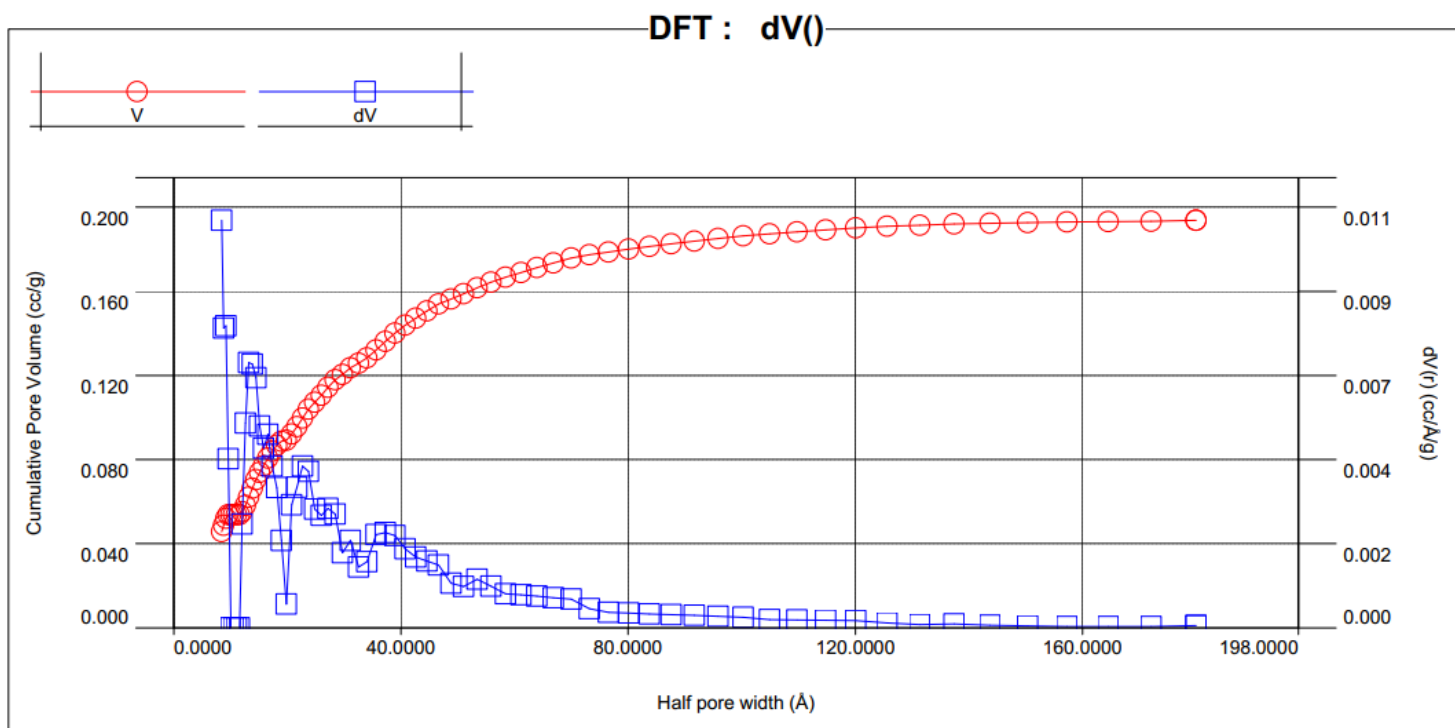


Figure 7

Figure 3.5. Surface area parameters by the DFT method

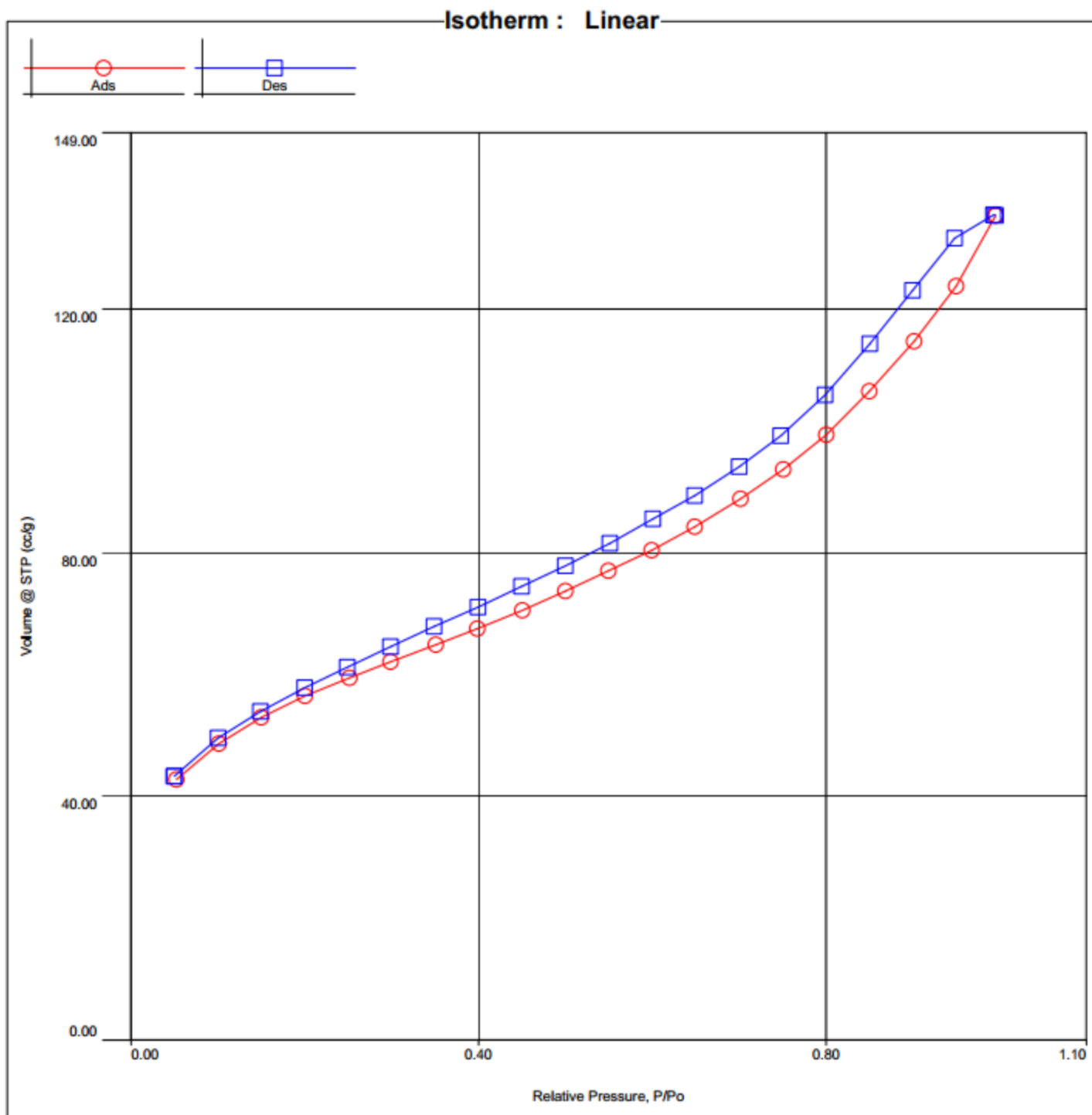


Figure 8

Figure 3.6. Isotherm: Linear

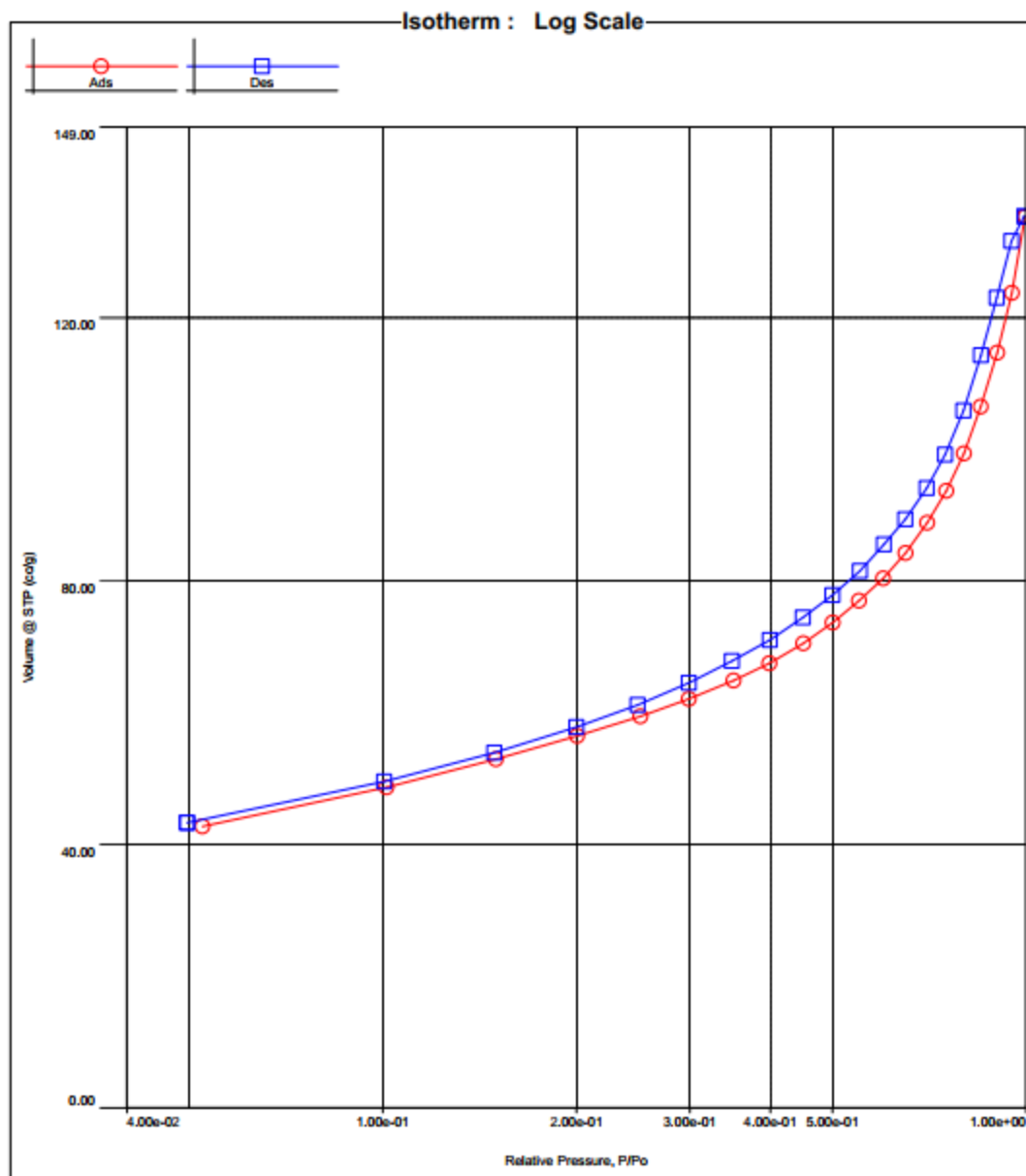


Figure 9

Figure 3.7. Isotherm: Log Scale

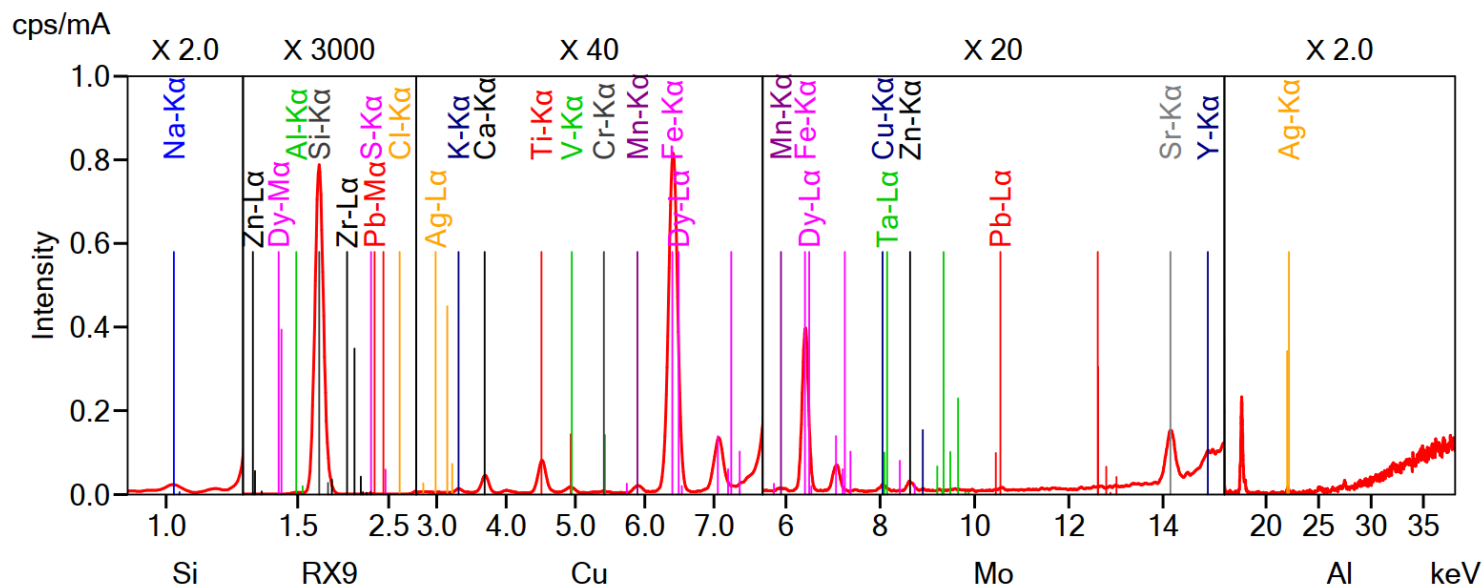


Figure 10

Figure 3.8. X-ray fluorescence (XRF) analysis spectrum of the obtained silica gel.