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# ILMIY AXBOROTNOMA

## НАУЧНЫЙ ВЕСТНИК
## SCIENTIFIC JOURNAL

## ANIQ VA TABIIY FANLAR SERIYASI

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# REMOVAL OF HEAVY METAL CATIONS USING MESOPOROUS SILICAGEL MODIFIED WITH L-CYSTEINE

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**Abstract.** Mesoporous $SiO_2$ was synthesized via sol-gel technology under acidic conditions at 50°C. The surface of the obtained sorbents was modified with the amino acid L-cysteine, and their textural characteristics were investigated. After modification with L-cysteine, the specific surface area increased to 975.6 $m^2/g$, and the nitrogen adsorption capacity reached 7.3 mol/kg. The adsorption of $Cu^{2+}$ and $Pb^{2+}$ cations on the sorbents was also studied. The adsorption capacities were found to be 0.41 mmol/l for $Cu^{2+}$ and 0.73 mmol/l for $Pb^{2+}$.

**Keywords:** mesoporous silica gel, adsorption capacity, Langmuir and Freundlich isotherms.

## L-sistein bilan modifikatsiyalangan mezog‘ovak silikagel yordamida og’ir metall kationlarini ajratish

**Annotatsiya.** 50°C da kislotali muhitda zol-gel texnologiyasi orqali mezog‘ovak $SiO_2$ sintez qilindi. Olingan sorbentlar sirti L-sistein aminokislotasi bilan modifikatsiyalandi va uning tekstur xarakterestikalari o‘rganildi. L-sistein bilan modifikatsiyalangandan so‘ng solishtirma sirti 975,6 $m^2/g$, azot bo‘yicha adsorbsiya sig‘imi esa 7,3 mol/kg ga ortishi kuzatildi. Shuningdek, sorbentlarga $Cu^{2+}$ va $Pb^{2+}$ kationlarining sorbsiyasi o‘rganildi. Unga ko’ra $Cu^{2+}$ adsorbilangan miqdori 0,41 mmol/l, $Pb^{2+}$ kationi esa 0,73 mmol*l* ekanligini aniqlandi.

**Kalit so‘zlar.** mezog‘ovak silikagel, adsorbsiya sig‘imi, Langmyuir va Freundlix izotermalari.

## Разделение катионов тяжелых металлов с помощью мезопористого силикагеля, модифицированного L-цистеином

**Аннотация.** Мезопористый $SiO_2$ был синтезирован с использованием технологии золь-гель при кислотной среде при температуре 50 °C. Поверхность полученных сорбентов была модифицирована аминокислотой L-цистеин, после чего были исследованы их текстурные характеристики. После модификации удельная поверхность увеличилась до 975,6 $м^2/г$, а емкость по адсорбции азота составила 7,3 моль/кг. Также была изучена сорбция катионов $Cu^{2+}$ и $Pb^{2+}$. Установлено, что количество адсорбированного $Cu^{2+}$ составило 0,41 ммоль/л, а для $Pb^{2+}$ — 0,73 ммоль/л.

**Ключевые слова:** мезопористый силикагель, сорбционная емкость, изотермы Лэнгмюра и Фрейндлиха.

**Introduction.** In recent years, the synthesis of a new class of organo/inorganic hybrid nanomaterials has rapidly increased. In the field of *materials science and the synthesis of new materials*, such compounds are obtained through molecular modeling, structural design, and surface modification with various functional groups. As a result, clusters, quantum dots, and supramolecular hybrid nanocomposites with unique physicochemical properties are being developed, playing an indispensable role in the advancement of technologies such as electronics, aerospace, medicine, pharmaceuticals, dentistry, and genetic engineering [1–3].

Among nanomaterials, mesoporous silica ($SiO_2$) holds special practical significance and is widely used in industrial and manufacturing sectors [4]. The effectiveness and applicability of porous silica materials depend greatly on their structural and surface morphological characteristics — including high specific surface area, pore sizes in the nanometer range, excellent biocompatibility, and distinct physicochemical features.

Typically, silica nanomaterials with pore sizes ranging from 0.4 to 50 nm and a high density of hydroxyl groups are of great importance in medicine and pharmacology. These porous nanomaterials are extensively applied in drug delivery (as carriers for molecules with various functional groups), in chromatographic analysis, enzyme immobilization, and even as binders for specific viruses [5–7]. Micro- and mesoporous hybrid organo/inorganic nanosorbents modified with diverse functional groups are of practical relevance in liquid and ion exchange chromatography for separation and analysis of complex mixtures. Notably, the synthesis of silica materials is not only of applied importance but also crucial for advancing fundamental theoretical research [8–10].

The silanol (Si–OH) groups on mesoporous silica nanoparticles serve as highly reactive and safe anchoring sites for organic functionalization [11–13]. To introduce certain organic groups, post-synthesis surface modification is generally applied to various forms of silica materials. In this study, the structure and electronic nature of a mesoporous silica–L-cysteine complex, obtained via sol-gel synthesis followed by post-synthetic modification with L-cysteine, were investigated using quantum chemical methods [14–15].

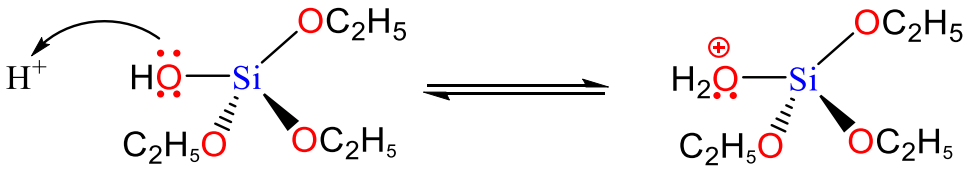
**Research Objective -** to synthesize mesoporous $SiO_2$ sorbents using sol-gel technology, modify their surfaces with the amino acid L-cysteine, evaluate their textural and sorption properties, and investigate the efficiency of the modified sorbents in the separation of $Cu^{2+}$ and $Pb^{2+}$ cations.

**Materials and Methods.** To synthesize mesoporous silica modified with L-cysteine, tetraethyl orthosilicate (TEOS, 96.8%) was used as the silica precursor. Ethanol (96.4%) served as the solvent, and the pore-forming structure-directing agent was included in the reaction mixture. Hydrochloric acid (0.01 M HCl) was used as the hydrolysis catalyst. The modifier L-cysteine, of analytical grade purity, was covalently anchored to the surface of mesoporous silica via post-synthesis functionalization.

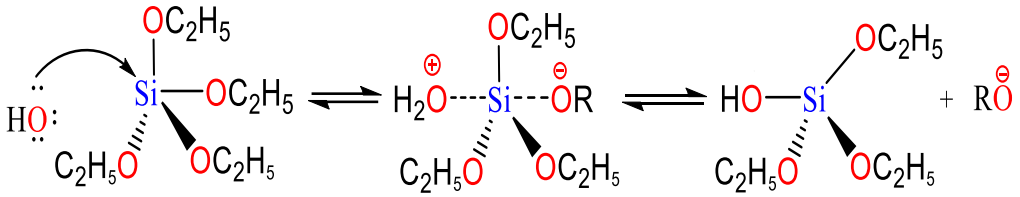
**Synthesis and Modification of Mesoporous $SiO_2$.** For the synthesis of the target complex, mesoporous $SiO_2$ nanoparticles were first obtained from TEOS using the sol-gel technology. Typically, hydrolysis reactions in the sol-gel process are carried out in the presence of an acid or base catalyst in an aqueous medium.

The synthesis was conducted at 50 °C using the reagents in the following molar ratio: TEOS: $C_2H_5OH$: $H_2O$: HCl = 1 : 4 : 4 : 0.01. Initially, 5 mL of a solution of the triblock copolymer P123 in ethanol was added to distilled water and stirred for 30 minutes until a homogeneous solution was formed. Then, under stirring with a magnetic stirrer, HCl was added dropwise until pH 2 was reached. Subsequently, the ethanol solution of TEOS was added dropwise over 10 minutes, and the mixture was stirred continuously for 12 hours until a transparent gel formed. The resulting gel was washed several times with distilled water and calcined at 500 °C to obtain mesoporous $SiO_2$.

In the sol-gel synthesis of mesoporous sorbent samples, the aqueous acid-catalyzed hydrolysis of tetraethoxysilane leads to the formation of a sol, which then undergoes a polycondensation reaction to form a gel. The general scheme of the acid-catalyzed hydrolysis of tetraethoxysilane is:



In acidic medium, the gel formation through the polycondensation reaction proceeds as follows:



To graft L-cysteine onto the surface of the synthesized mesoporous silica, the dried powder was immersed in an aqueous solution of L-cysteine and kept for three days. Afterward, the water was evaporated, resulting in L-cysteine-modified mesoporous silica sorbents. The overall synthesis and modification process of mesoporous silica via sol-gel and L-cysteine functionalization is illustrated in **Figure 1**.

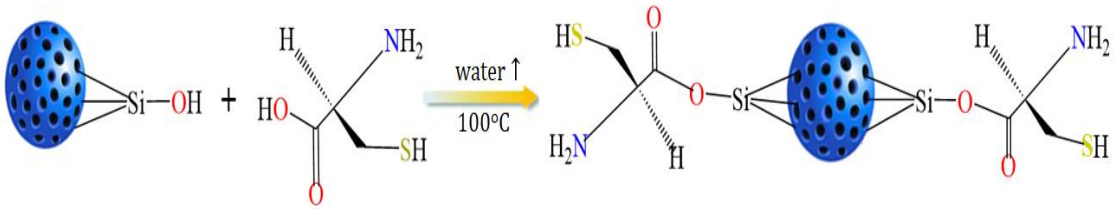



**Figure 1.** Modification of mesoporous silica surface with L-cysteine

**Results and discussion.** The surface morphology and pore size of the synthesized mesoporous sorbent were studied using scanning electron microscopy (SEM) with a Carl Zeiss EVO MA 10 electron microscope, equipped with an energy-dispersive X-ray spectrometer (EDS Aztec Energy Advanced X-Act, Oxford Instruments). The analysis was performed under an accelerating voltage up to 1 kV, with magnifications up to ×200,000, using secondary electron (SE2) and backscattered electron (ESB) detectors.

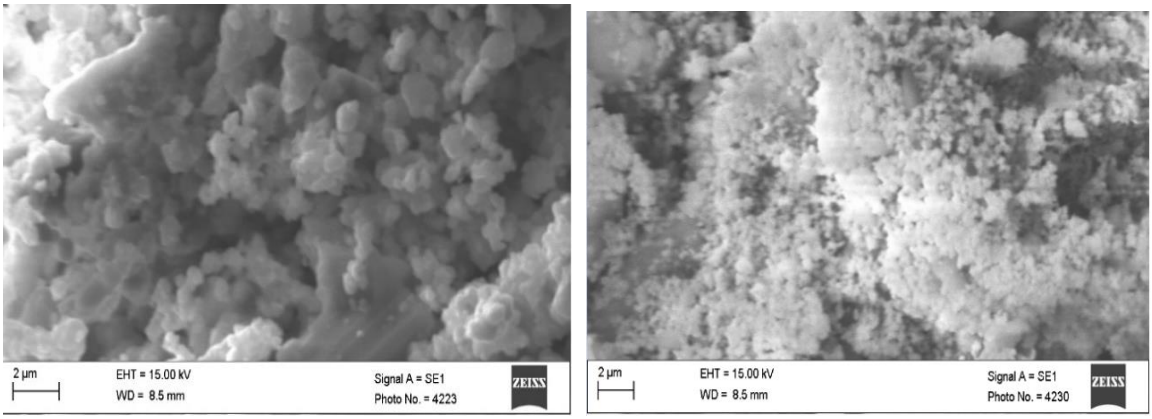



Figure 2. SEM Images of Mesoporous Silica Gel and L-Cysteine-Modified Sorbents

From the SEM images, it can be observed that the surface of the synthesized sorbents exhibits monodispersity with spherical $SiO_2$ nanoparticles. After modification with L-cysteine, changes in the surface morphology of the mesoporous $SiO_2$ can be clearly seen in Figure 2b, indicating successful surface functionalization.

The textural characteristics of both unmodified and L-cysteine-modified mesoporous $SiO_2$ were investigated using nitrogen vapor adsorption analysis. In both cases, the adsorption isotherms showed a sharp increase at relative pressure $p/p_0 < 0.2$, followed by saturation near $p/p_0 = 0.9$. Between $p/p_0 = 0.4–0.8$, the adsorption and desorption curves diverged, forming a hysteresis loop, which indicates capillary condensation in mesopores.

This behavior confirms that the sorbent samples are mesoporous and belong to Type IV isotherms according to the IUPAC classification. Furthermore, it was observed that the nitrogen adsorption capacity of the sorbent increased after modification with L-cysteine.

The adsorption data at different relative pressures were fitted to the linear form of the BET equation, showing a correlation coefficient of $R^2 = 0.997$ (Figure 3b), indicating excellent agreement and reliability of the BET analysis results.

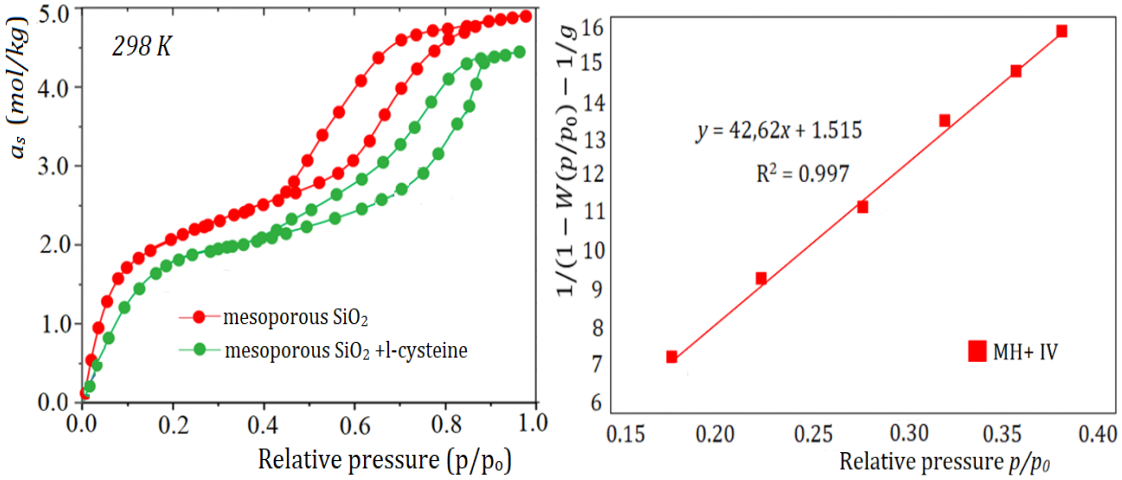



Figure 3. Adsorption Isotherms of Mesoporous $SiO_2$ and Modified Sorbent

Using the adsorption isotherms and the linear form of the BET equation, key textural characteristics of the sorbents obtained at different temperatures were calculated, including: specific surface area ($S_{BET}$), pore diameter ($D$, *nm*), monolayer adsorption capacity ($a_m$), and saturation adsorption capacity ($a_s$).

Table

Textural Characteristics of the Sorbents

| Sorbent | $S_{BET}$, $m^2/g$ | $a_m$, mol/kg | D, nm | $a_s$, mol/kg |
|---|---|---|---|---|
| Mesoporous $SiO_2$ | 612,2 ± 100 | 1,6 ± 0,5 | 6,5 ± 1,18 | 4,0 ± 0,8 |
| Mesoporous $SiO_2$ modified with L-cysteine | 975,6 ± 150 | 2,3 ± 0,7 | 16,8 ± 0,05 | 7,3 ± 1,2 |

It can be observed from the table that after modification of mesoporous $SiO_2$ with L-cysteine, both the surface area and the nitrogen adsorption capacity increase.

The obtained sorbent samples were used to separate $Cu^{2+}$ and $Pb^{2+}$ cations from wastewater. For this purpose, adsorption of the metal cations from a 10 mL solution onto the powdered sorbent was carried out. The amount of metal ion adsorbed onto the sorbent was calculated using the following formula:

$$q_e = \frac{(C_0 - C_e) \cdot V}{m} \quad (1)$$

Where $q_e$ – amount of metal cations adsorbed (mg/g), $C_0$ – initial concentration of the metal cations in the solution (mmol/L), $C_e$ – equilibrium concentration (mmol/L), m – mass of the adsorbent (g), V – volume of the solution (L).

Using equilibrium adsorption data, the amount of metal ions adsorbed (Q, mmol/L), adsorption efficiency ($a$, %), and equilibrium constants for the L-cysteine modified mesoporous $SiO_2$ were calculated and summarized in **Table 2**. The equilibrium (2) constant for the adsorption process was calculated based on the following equation:

$$ln\ (q_e - q_t = \frac{(ln\ (q_e) - K_{1p}}{2{,}303}. (2)$$

**Table 2**

*Adsorption data of metal cations on modified mesoporous $SiO_2$*

| Metal cation | $C_0$ (mmol· $l$) | $C_e$ (mmol· $l$) | Q, (mmol· $l$) | $a$ (%) | $K_d$ (mL $g^{-1}$) |
|---|---|---|---|---|---|
| $Cu^{2+}$ | 2,23 | 0,015 | 0,71 | 98,64 | 3,9800 |
| $Pb^{2+}$ | 4,2 | 0,35 | 0,43 | 92,82 | 2,086 |

Furthermore, the adsorption of metal cations onto both the unmodified and L-cysteine modified mesoporous $SiO_2$ synthesized via the sol-gel method was modeled using Langmuir and Freundlich isotherms (Figure 4).

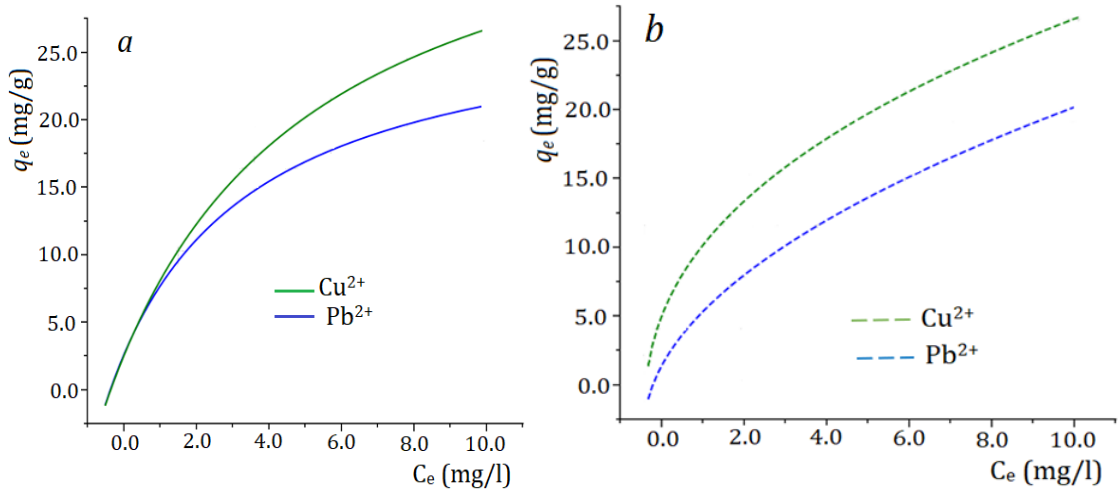



Figure 4. Langmuir and Freundlich isotherms for the adsorption of metal cations on modified mesoporous $SiO_2$

From the graphs, it is evident that after modification of mesoporous $SiO_2$, the adsorption capacity increased. The Langmuir isotherm graph represents the monolayer adsorption of $Cu^{2+}$ and $Pb^{2+}$ ions onto the surface. It shows that the adsorption of $Cu^{2+}$ ions is higher than that of $Pb^{2+}$, which is related to the stronger interaction between the sorbent and the $Cu^{2+}$ ion. As the equilibrium concentration increases, the adsorption amount gradually reaches saturation.

The Freundlich isotherm graph represents multilayer and heterogeneous surface adsorption of $Cu^{2+}$ and $Pb^{2+}$ ions on the modified mesoporous $SiO_2$ sorbent. It also shows that the adsorption of $Cu^{2+}$ ions is higher than that of $Pb^{2+}$ ions. The continued increase in adsorption with rising concentration indicates that the surface contains sites with varying energy levels. This model provides an empirical description of the sorption process.

**Conclusions.** Modification of mesoporous $SiO_2$ synthesized by the sol-gel method with the amino acid L-cysteine significantly improved the textural properties of the sorbent — the specific surface area increased up to 975.6 $m^2/g$, and the adsorption capacity reached 7.3 mol/kg.

1. The modified sorbents demonstrated high sorption activity toward $Cu^{2+}$ and $Pb^{2+}$ cations, with adsorption amounts of 0.41 mmol/L and 0.73 mmol/L, respectively. This makes them effective for the separation and removal of heavy metal ions.

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