ADSORPTION REMOVAL OF Cu (II) USING Ni-MODIFIED SILICA GEL

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The contamination of surface and groundwater with heavy metals represents a significant threat to environmental safety and the health of living organisms. Consequently, the development of new technologies and the improvement of existing ones to clean water bodies from toxicants of various kinds represents an important task. One of the factors influencing the choice of treatment method is the concentration of the pollutant. It is advisable to use adsorption methods for the post treatment of water environmental to the levels of maximum permissible concentrations. In the context of the implementation of adsorption methods, the selection of sorbents that combine high efficiency and low cost with ease of preparation represents an important factor. The aim of this work is to produce a sorbent based on commercial silica gel and nickel oxide. For its synthesis, the thermolysis method was chosen. A series of materials with varying mass ratios of NiO to SiO$_2$ was produced: 1:1 and 0.5:1. The presence of crystalline nickel oxide on the amorphous surface of SiO$_2$ was confirmed by X-ray diffraction analysis. The main parameters of the porous structure were determined by the method of low-temperature nitrogen adsorption/desorption. With an increase in the amount of the deposited oxide layer, the specific surface area and pore volume of the obtained samples decrease by 1.5-2.5 times compared to the original SiO$_2$. At the same time, the general character of the mesoporous structure is preserved. The optimal conditions for the removal of Cu (II) ions by nickel-containing composites based on silica gel (pH ~ 5.5; sorption time - 1 hour) were investigated. The adsorption kinetics is adequately described by a pseudo-second-order model, which indicates a high affinity of copper for the surface of such samples. It was demonstrated that the modification of the SiO$_2$ surface with nickel oxide results in an enhancement of the sorption capacity of materials with respect to Cu (II) ions. The maximum value of copper sorption on the synthesised samples is 10 times higher than on the original SiO$_2$.

Keywords: *adsorption, copper, modification, nickel oxide, silica gel, water treatment*

1. Introduction

Water is essential to life and seems to be everywhere and inexhaustible. In fact, however, more than 1.1 billion people worldwide are suffering from a shortage of clean water (Mishra, 2023). The deterioration of water bodies can be attributed to the advent of industrialisation, which has led to the contamination of these ecosystems by a multitude of wastewaters generated by human activities. These pollutants enter water bodies through a multitude of pathways, including direct discharge, runoff, and atmospheric deposition. One of the most pressing problems is the pollution of heavy metals in water bodies. If wastewater containing heavy metals is discharged directly without control, the heavy metals in water bodies that are not easily biodegradable will continue to be enriched through the biological chain, and

ultimately irreversible damage to human health will occur (Aziz, 2023; Zamora-Ledezma, 2021).

Different chemical properties of heavy metals necessitate optimal ways to remove them from wastewater. The current methods used to treat wastewater from heavy metal ions include chemical, physical, and biological ones: precipitation, ion exchange, adsorption, membrane filtration, coagulation, flocculation, flotation, and electrochemical methods (Kovalchuk, 2023).

The method of pollutant removal is dependent on the form of the metal in question, its concentration, the content of salt and organic matter in natural or wastewater, and the effectiveness of the chosen method. The most effective purification of water from heavy metal ions is achieved through adsorption methods, which are able to remove metal ions from water at very low concentrations compared to other methods (Kholodko, 2022).

Now there are many adsorbents of various nature and origin for the use in removal wastewater technologies (Bondarieva, 2023; Pylypenko, 2023). In particular, it has been found that metal oxide-based adsorbents have unique physicochemical properties, including chemical stability, controllable morphology and size, and abundant surface-active sites. They have potential applications in the removal of various heavy metal contaminants from water systems (Wang, 2020; Gupta, 2021). For example, iron oxide (Fe_3O_4), which is common among metal oxides, has been shown to be effective in the removal of various heavy metals (including Pb (II), Cd (II), Cu (II), Cr (VI)) due to its magnetic properties and ease of separation from the adsorbed wastewater (Fayyazi, 2023; Shen,

2020; Tao, 2021). Another example includes metal oxide composites using titanium dioxide (TiO_2), which is well known for its large specific surface area and stable properties (Chen, 2018; Siddeeg, 2020). In addition, nickel oxide (NiO) stands out for its excellent physicochemical properties, with its high specific surface area, low cost, and natural porosity showing great potential for water treatment (Behnajady, 2014; Bhardwaj, 2020; Hashem, 2022). These examples highlight that metal oxide nanoparticles and their complexes possess the potential to act as effective adsorbents for water purification.

In view of these findings, our study aimed to design a silica-based composite embedded with nickel oxide (NiO) particles. This was based on several considerations. First, it is well known that the surface of SiO_2 is negatively charged in water, while the surface of NiO is positively charged, which allows the two to bond uniformly and tightly together due to charge attraction (Rubab, 2021). Silicon dioxide material serves as an excellent support substrate, and its chemical stability and high specific surface area can provide nickel oxide particles with a substrate bed that is stable and reactive in a variety of environments and can control the size of NiO particles to a certain extent, as well as enhance their dispersion and heavy metal adsorption capacity. In addition, NiO was selected for its special affinity for heavy metal ions, and the literature shows that it still maintains good adsorption performance in aqueous environments where a variety of heavy metal ions are present (Dhiman, 2021). Therefore, modification of silica surface with nickel oxide particles can synergize the properties of the two materials, thus significantly improving the adsorption performance. The adsorption capacity of the

obtained materials was evaluated by purifying water from copper ions.

2. Materials and Methods

2.1 Chemicals and reagents

The following chemicals and reagents were used in this work: commercial high-purity grade silica gel (SiO_2) with a particle size of 230-400 mesh, nickel nitrate ($Ni(NO_3)_2 \cdot 6H_2O$), 65% nitric acid (HNO_3) and Sucrose (99.9%) were purchased from Sigma-Aldrich (USA), NaOH (Ineos Group, France), NaCl (Brenntag SE, Germany). In all studies conducted, distilled water was utilized as the solvent.

2.2 Method for preparation of NiO-modified samples

The materials were synthesized according to a modified method described in (El-Quanni, 2017). In the first stage, a 2 wt.% aqueous suspension of SiO_2 was prepared. Then, while continuing to stir it with a magnetic stirrer, the calculated amount of $Ni(NO_3)_2 \cdot 6H_2O$ and 13 g of sucrose were added sequentially. After complete dissolution, 1.5 ml of HNO_3 was added and the system was left to stir for 30 min at room temperature. The calculation of the exact weight of $Ni(NO_3)_2 \cdot 6H_2O$ was performed with the need to maintain the mass ratio of SiO_2:NiO = 1:1 and 1:0.5.

The next stage of the synthesis consists of successive temperature treatment stages at 110 °C, 250 °C, and 800 °C using a drying cabinet (Memmert UNB300, Germany) and a muffle furnace (SNOL-1.6.2.5.1/9-I4, Latvia). After aging at 110 °C, the samples are in the form of biscuit cakes. A temperature of 800 °C is responsible for the implementation of the thermolysis process in the air atmosphere with the following conditions:

heating rate of 7 °C/min, holding at a maximum temperature of 3 hours. For further studies, the fired samples were crushed and sieved to obtain a fraction of ≤ 0.315 mm.

2.3 Characterization and analytic methods

The successful modification of the surface of amorphous SiO_2 was confirmed by identifying the NiO crystal phase by X-ray diffraction using a DRON-4 (Bourevestnik, USSR) with CuKα-radiation, step-size 0.1° and a holding time of 3 seconds. The crystal structure of the samples was analyzed using the JCPDS Database (International Center for Diffraction Data).

The characteristics of the porous structure were determined by the method of low-temperature nitrogen adsorption-desorption (Quantachrome NOVA-2200, USA). The data obtained were processed using ASIQwin software. The specific surface area (S_{BET}, m^2/g) was calculated by the Brunauer-Emmett-Teller (BET) multipoint method. The total pore volume (V_Σ, cm^3/g) was determined by the maximum adsorbed volume of nitrogen at a relative pressure $p/p_0 \approx 1$. The Barrett-Joyner-Halenda (BJH) model was used to determine the pore size distribution.

The adsorption capacity of the obtained samples was evaluated using model solutions contaminated with Cu (II) ions in the concentration range of 0.1-25 mg/dm^3. They were prepared in distilled water using a standard copper solution with a concentration of 1000 mg/dm^3 (Sigma-Aldrich, USA). The ionic strength (I=0.01) was adjusted with 1M NaCl solution. The pH value was adjusted with 0.1M NaOH solution and controlled with a pH meter (ADWA AD1020, Hungary).

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To determine the optimal conditions for the purification of model solutions from Cu (II) ions, we additionally investigated the effect of the pH of the medium and the duration of contact of the adsorbent with the contaminated water on the efficiency of toxicant removal.

The analysis of the parameters of kinetic processes was carried out using pseudo-first-order (PFO) and pseudo-second-order (PSO) models (Eq. 1 and Eq. 2, respectively):

$$l\,n(a_e - a_t) = lna_e - k_1 t \qquad (1)$$

$$\frac{t}{a_t} = \frac{1}{k_2 a_e^2} + \frac{t}{a_e} \qquad (2)$$

where a_e and a_t (mg/g) are the adsorption capacity at equilibrium and at any time (min), respectively; k_1 (min^{-1}) and k_2 (g/mg·min) are the PFO and PSO rate constant, respectively.

All adsorption experiments were carried out under static conditions at a temperature of 20 ± 2 °C and continuous shaking of the samples (Biosan OS-20, Latvia) for 1 hour (aqueous phase volume 50 cm^3, sorbent weight 0.1 g). After the adsorption equilibrium was established, the liquid phase was separated by centrifugation (3600 rpm) and the equilibrium concentration of copper was determined by inductively coupled plasma atomic emission spectrometry (Thermo Scientific iCAP 7400 ICP-OES, USA).

The adsorption capacity (a, mg/g) and removal efficiency (X, %) of copper ions were estimated by Eq. (3) and Eq. (4), respectively:

$$a = \frac{(C_{in} - C_{eq}) \cdot V}{m} \qquad (3)$$

$$X\,(\%) = \frac{C_{in} - C_{eq}}{C_{in}} \cdot 100 \qquad (4)$$

where C_{in} and C_{eq} are representing the initial and equilibrium copper (II) ions concentrations (mg/md^3), V is the solution volume (dm^3), and m is the weight of adsorbent (g).

3. Results and discussion

When analyzing the XRD (X-Ray Diffraction Analysis) data for the SiO$_2$@NiO sample (Fig. 1), 5 characteristic peaks were observed at 2θ = 37°, 43°, 62°, 75°, and 79°, corresponding to the planes (222), (400), (440), (622), (444), respectively, and belonging to NiO (JCPDS database card No. 89-5881). The broad peak at $2\theta \approx 22°$ is characteristic of SiO$_2$ in the amorphous phase. The SiO$_2$@0.5NiO sample has a similar X-ray diffraction pattern, but the intensity of the main characteristic peaks is lower.
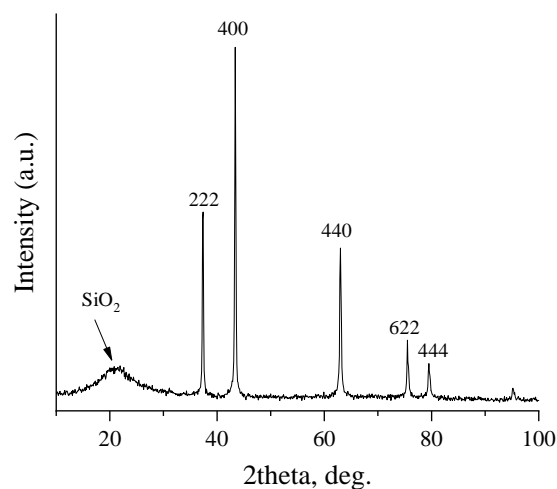

Fig. 1. *Diffraction pattern of the synthesized SiO$_2$@NiO*

The obtained isotherms of low-temperature nitrogen adsorption/desorption (Fig. 2) belong to the type IV isotherms according to the IUPAC classification.
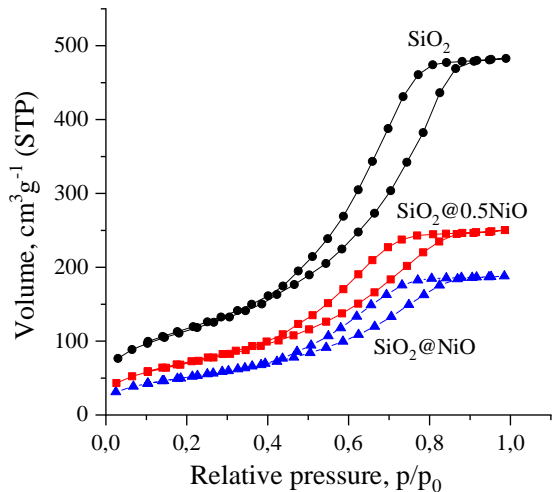
Fig. 2. *Isotherms of low-temperature N$_2$ adsorption/desorption of the pure and modified SiO$_2$*

This type of isotherms is typical for materials with a mesoporous structure.

The nature of the hysteresis loop allows us to determine that the porous structure of all samples is formed by spherical particles of similar size with homogeneous packing, forming cylindrical pore channels. Also, these materials have a narrow range of homogeneous mesopores concentrated around 2.5 – 3 nm, which is confirmed by the distribution of pores by radius (Fig. 3).
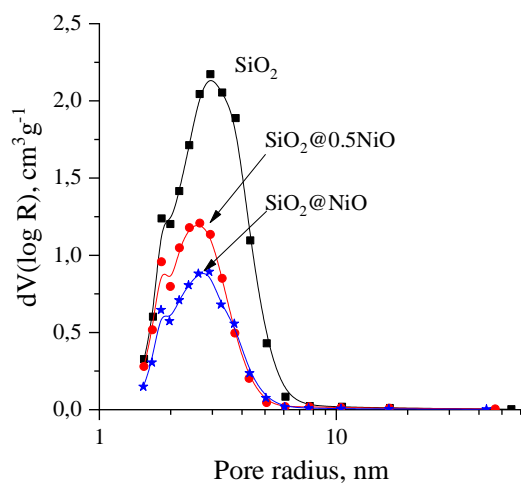

Fig. 3. *Pore size distribution by radius of the pure and modified SiO$_2$*

The parameters of the porous structure of the investigated samples are presented in Table 1.

Table 1. *Parameters of the porous structure of SiO$_2$ samples before and after Ni-modified*

Sample	S_{BET}, m^2/g	V_Σ, cm^3/g	Pore radius dV(r), nm
SiO$_2$	411	0.746	2.653
SiO$_2$@0.5NiO	256	0.387	1.830
SiO$_2$@NiO	186	0.290	1.822

Note: S_{BET} – specific surface area, V_Σ – total pore volume, dV(r) – average pore radius (BJH).

With an increase in the amount of the deposited NiO layer on the SiO$_2$ surface, the value of the specific surface area decreases by 1.5-2.5 times. In addition, the total volume of pores decreases, indicating that they are partially blocked by NiO particles or that NiO agglomerates are formed on the pore walls.
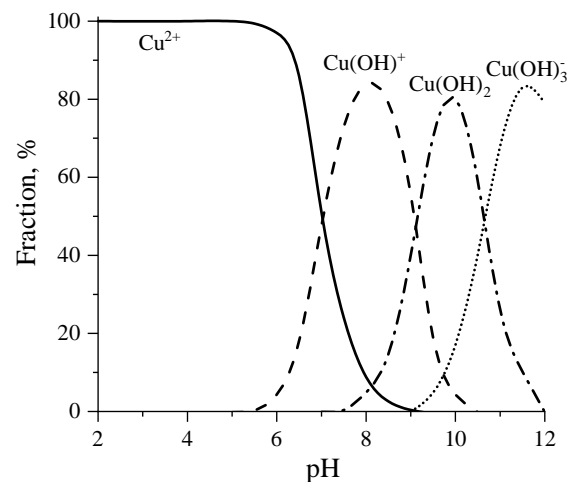

Fig. 4. *Diagram of the distribution of copper forms depending on pH of the aqueous solution*

It is well established that the initial pH of a solution affects not only the protonation

of the functional groups of adsorbents, but also the degree of ionization and speciation of toxicants in an aqueous solution. This, in turn, affects the interactions between the adsorbents and the adsorbate. Fig. 4 shows the distribution of copper ion forms in aqueous medium depending on pH (calculated using the software "The software Chemical Equilibrium Diagrams", Sweden).

In an acidic environment (pH<6), copper is found mainly in the form of positively charged Cu^{2+} ions. When the pH shifts towards an alkaline environment, copper is precipitated in the form of hydroxides. The dynamics of this process depends on the concentration of copper, the presence of other anions and cations, and temperature. Accordingly, in natural aqueous systems, copper hydroxocomplexes can be formed in the pH range of 6.5 and higher (Cuppett, 2006). Therefore, the effect of pH on the adsorption of Cu (II) was studied in the pH range from 3 to 5.5 to avoid the precipitation of copper in the form of $Cu(OH)_2$ at pH > 6.

According to the results obtained, commercial SiO_2 shows almost no sorption capacity for Cu (II) in the studied pH range, and the maximum sorption value is 0.2 mg/g at pH 5.5. For the modified samples, the degree of Cu^{2+} ion removal increases simultaneously with an increase in pH. Thus, the maximum sorption values are observed at pH 5.5 and are equal to 0.9 mg/g and 1.7 mg/g for the SiO_2@0.5NiO and SiO_2@NiO samples, respectively. That is why all subsequent sorption experiments were performed at pH 5.5±0.1.

The study of the influence of the contact time between the samples modified with nickel oxide and copper ions on the sorption

process showed that their removal occurs quite quickly (Fig. 5).

The time for establishing sorption equilibrium in the system is 1 hour. At the same time, within the first 15 minutes, the degree of removal for the SiO_2@NiO sample is 51%. At the same time, the SiO_2@0.5NiO sample removes only 36% of copper ions, which is much higher than unmodified SiO_2.

The analysis of the kinetics of copper ion sorption by the obtained sorbents was carried out using pseudo-first-order and pseudo-second order kinetic models. The calculated parameters of the kinetic models are shown in Table 2. The obtained data indicate that the kinetic dependences of Cu (II) sorption are better described by the pseudo-second-order model, since the calculated values of sorption values ($a_{e,\ cal,}$) coincide with the experimental data ($a_{e,\ exp}$) and the correlation coefficient is close to 1 ($R^2 > 0.99$).

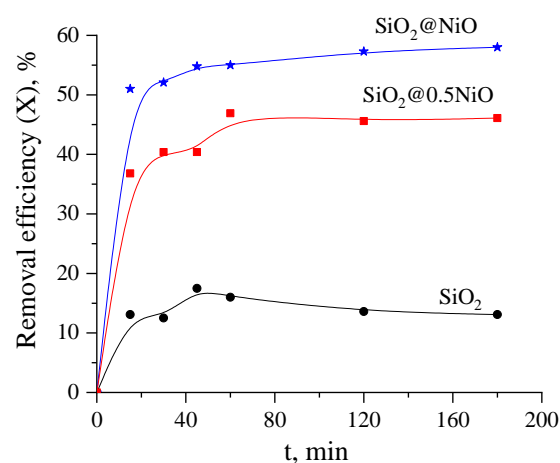***Fig. 5***. *Kinetic curves of Cu (II) adsorption on the studied materials*

Table 2. *Kinetic parameters of Cu (II) adsorption on Ni-modified silica gel*

Sample	$a_{e, exp}$, (mg/g)	Parameters					
		Pseudo-first-order			Pseudo-second-order		
		$a_{e, cal}$, (mg/g)	k_1, min^{-1}	R^2	$a_{e, cal}$, (mg/g)	k_2, g/mg min	R^2
SiO$_2$@0,5NiO	1.20	0.53	0.0085	0.554	1.20	0.2158	0.997
SiO$_2$@NiO	1.34	0.37	0.0192	0.786	1.33	0.3113	0.999

Fig. 6 shows the sorption isotherms of copper ions on pure commercial SiO$_2$, and modified samples based on it.
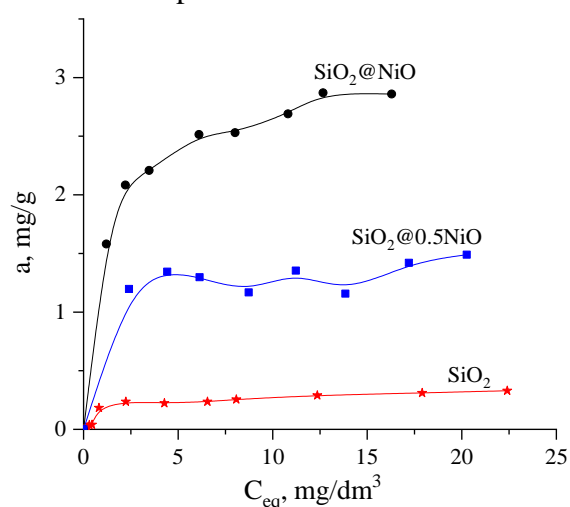

Fig. 6. *Isotherms of adsorption of Cu (II) by the pure and modified SiO$_2$ samples*

The results obtained indicate that the sorption capacity of the original SiO$_2$ in relation to copper ions is insignificant and reaches no more than 0.3 mg/g.

At the same time, after modifying its surface with nickel oxide, an increase in this indicator is observed. Thus, for the SiO$_2$@0.5NiO sample, the increase in the maximum sorption value is almost 5 times, and for SiO$_2$@NiO, almost 10 times.

4. Conclusions

A range of materials, based on commercia silica gel and nickel oxide, was synthesised with differing mass ratios of the components. The analysis of the textural characteristics showed that the obtained samples retained the mesoporous structure of the original matrix, although the average pore radius a slight decline. Furthermore, in the series SiO$_2$ > SiO$_2$@0.5NiO > SiO$_2$@NiO, the specific surface area decreased from 411 m^2/g to 186 m^2/g.

The adsorption characteristics of these materials exhibit an inverse trend. That is, in the concentration range of 0.1-25 mg/dm^3, a maximum sorption value is observed for the SiO$_2$@NiO sample, with a value of 2.86 mg/g, which is 10 times higher than the value of adsorption of unmodified SiO$_2$.

The removal of copper ions is quite fast. For instance, for the sample with the maximum NiO coating, it can be observed that the degree of Cu (II) removal is more than 50% within the initial 15 minutes. The kinetic process is described by a pseudo-second-order model.

In future studies, it would be interesting to investigate the adsorption capacity of Ni-modified silica materials for organic toxicants.

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АДСОРБЦІЙНЕ ВИДАЛЕННЯ Cu (II) З ВИКОРИСТАННЯМ Ni-МОДИФІКОВАНОГО СИЛІКАГЕЛЮ

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Забруднення поверхневих та підземних вод важкими металами становить серйозну загрозу для екологічної безпеки та здоров'я живих організмів. Саме тому, актуальним завданням є розробка нових і вдосконалення існуючих технологій для очищення водних об'єктів від токсикантів різної природи. Одним з факторів, що впливає на вибір методу очищення є концентрація забруднювача. При доочищенні водних середовищ до рівнів гранично допустимих концентрацій доцільно використовувати адсорбційні методи. Для їх успішної реалізації, важливим є використання сорбентів, що поєднують високу ефективність з низькою вартістю та простотою одержання. Метою даної роботи є отримання сорбенту на основі комерційного силікагелю та нікель (II) оксиду. Для синтезу якого було обрано метод термолізу. Отримано серію матеріалів з різним масовим співвідношенням NiO до SiO₂: 1:1 та 0.5:1. За допомогою рентгенофазового аналізу підтверджено наявність кристалічного оксиду нікелю на аморфній поверхні SiO₂. Методом низькотемпературної адсорбції/десорбції азоту визначено основні параметри пористої структури. Так, при збільшенні кількості нанесеного оксидного шару питома поверхня та об'єм пор отриманих зразків зменшуються в 1,5-2,5 рази у порівнянні з вихідним SiO₂. При цьому загальний характер мезопористої структури зберігається. Досліджено оптимальні умови вилучення іонів Cu (II) нікельвмісними композитами на основі силікагелю (pH ~ 5,5; тривалість сорбції – 1 година). Кінетика адсорбції адекватно описується моделлю псевдо-другого порядку, що вказує на високу спорідненість купруму з поверхнею таких зразків. Встановлено, що модифікування поверхні SiO₂ нікель (II) оксидом призводить до підвищення сорбційної здатності матеріалів по відношенню до іонів Cu (II). Максимальна величина сорбції Cu (II) на синтезованих зразках в 10 разів вища ніж на вихідному SiO₂.

Ключові слова: *адсорбція, купрум, модифікування, нікель (II) оксид, очищення вод, силікагель*