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EVALUATION OF THE SYNERGETIC EFFECT OF PYRENEE SORPTION OF CU(II) IONS BASED ON LANGMUIR-FREUNDLIC MODELING

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Abstract. This study investigates the adsorption properties of pyrene in the absence and presence of Cu(II) ions over a wide concentration range (log Ce = 0.0–0.1). In the Cu-free system (0 mg L^{-1}), the adsorption capacity increased from 9 mg g^{-1} at log Ce = 0.0 to 52 mg g^{-1} at log Ce = 0.1. The intermediate values were 15, 22, 25, 33, and 39 mg g^{-1} at log Ce = 0.001, 0.005, 0.01, 0.025, and 0.05, respectively. In contrast, in the presence of 20 mg L^{-1} Cu(II), the sorption of pyrene increased significantly from 37 mg g^{-1} to 83 mg g^{-1} over the same concentration range. The adsorption capacities at the intermediate points reached 54, 58, 65, 67 and 74 mg g^{-1}, which showed a strong increase. The maximum increase in adsorption capacity was about 31 mg g^{-1}, and the minimum increase was about 28 mg g^{-1}. The relative adsorption enhancement varied from 1.6 to 3.6 times depending on the equilibrium concentration. These results indicate a significant synergistic effect between Cu(II) ions and pyrene molecules. The enhanced adsorption is attributed to the coordination-assisted π–π interactions and increased surface activity. Overall, the system exhibits high sorption efficiency and reaches a maximum capacity of 83 mg g^{-1} during the Cu-assisted adsorption process.

Keywords: Heavy metal sorption, competitive adsorption, Langmuir isotherm, Freundlich isotherm, adsorption capacity, surface chemistry modification, synergistic adsorption effect.

Cu(II) ionlarining Piren sorbsiyasini sinergetik ta’sirini Langmuir-Freundlich modellashtirish asosida baholash

Annotatsiya. Ushbu tadqiqot keng konsentratsiya oralig‘ida (log Ce = 0.0–0.1) Cu(II) ionlari yo‘qligida va mavjudligida pirenning adsorbsiya xususiyatini o‘rganadi. Cu-tizimda (0 mg L^{-1}), adsorbsiya sig‘imi log Ce = 0.0 da 9 mg g^{-1} dan log Ce = 0.1 da 52 mg g^{-1} gacha oshdi . Oraliq qiymatlar log Ce = 0.001, 0.005, 0.01, 0.025 va 0.05 da mos ravishda 15, 22, 25, 33 va 39 mg g^{-1} ni tashkil etdi. Aksincha, 20 mg L^{-1} Cu(II) mavjudligida, pirenning sorbsiyasi bir xil konsentratsiya oralig‘ida 37 mg g^{-1} dan 83 mg g^{-1} gacha sezilarli darajada oshdi. Oraliq nuqtalardagi adsorbsiya sig‘imlari 54, 58, 65, 67 va 74 mg g^{-1} ga yetdi , bu kuchli o‘sishni ko‘rsatdi. Adsorbsiya sig‘imining maksimal o‘sishi taxminan 31 mg g^{-1} ni tashkil etdi , minimal o‘sish esa taxminan 28 mg g^{-1} ni tashkil etdi . Nisbiy adsorbsiya yaxshilanishi muvozanat konsentratsiyasiga qarab 1,6 dan 3,6 martagacha o‘zgarib turdi. Bu natijalar Cu(II) ionlari va piren molekulalari o‘rtasida sezilarli sinergetik ta'sirni ko‘rsatadi. Kuchaygan adsorbsiya koordinatsiya yordamidagi π–π o‘zaro ta'sirlar va sirt faolligining oshganligini ko‘rsatadi. Umuman olganda, tizim yuqori sorbsiya samaradorligini namoyish etadi va Cu yordamidagi adsorbsiya jarayonida maksimal 83 mg g^{-1} sig‘imga erishadi.

Kalit so‘zlar: Og‘ir metallarni sorbsiyasi, raqobatbardosh adsorbsiya, Langmuir izotermasi, Freundlix izotermasi, adsorbsiya sig‘imi, sirt kimyosi modifikatsiyasi, sinergetik adsorbsiya effekti.

Оценка синергетического эффекта ионов Cu(II) на сорбцию пирена на основе моделирования Лангмюра-Фройндлиха

Аннотация. В данном исследовании изучаются адсорбционные свойства пирена в отсутствие и присутствие ионов Cu(II) в широком диапазоне концентраций (log Ce = 0,0–0,1). В системе без Cu (0 мг/л) адсорбционная способность увеличилась с 9 мг/г при log Ce = 0,0 до 52 мг/г при log Ce = 0,1. Промежуточные значения составили 15, 22, 25, 33 и 39 мг/г при log Ce = 0,001, 0,005, 0,01, 0,025 и 0,05 соответственно. В отличие от этого, в присутствии 20 мг/л Cu(II) сорбция пирена значительно увеличилась с 37 мг/г до 83 мг/г в том же диапазоне концентраций. Адсорбционная способность в промежуточных точках достигала 54, 58, 65, 67 и 74 мг·г$^{-1}$, демонстрируя значительное увеличение. Максимальное увеличение адсорбционной способности составило около 31 мг·г$^{-1}$, а минимальное — около 28 мг·г$^{-1}$. Относительное увеличение адсорбции варьировалось от 1,6 до 3,6 раз в зависимости от равновесной концентрации. Эти результаты указывают на значительный синергетический эффект между ионами Cu(II) и молекулами пирена. Усиленная адсорбция объясняется координационно-опосредованными π–π взаимодействиями и повышенной поверхностной активностью. В целом, система демонстрирует высокую эффективность сорбции и достигает максимальной емкости 83 мг·г$^{-1}$ в процессе адсорбции с участием Cu.

Ключевые слова: сорбция тяжелых металлов, конкурентная адсорбция, изотерма Лангмюра, изотерма Фройндлиха, адсорбционная способность, модификация поверхностной химии, синергетический эффект адсорбции.

1. **Introduction.** In the last two decades, the rapid development of industries such as metallurgy, mining, electrical engineering, and mechanical engineering has led to an increase in the content of metal ions such as lead (Pb), cadmium (Cd), mercury (Hg), chromium (Cr), nickel (Ni), and copper (Cu) in wastewater [1-4]. The high biological toxicity of these heavy metal ions, their ability to penetrate biostructures, and their ability to bioaccumulate in living organisms and have a long-term toxic effect make them priority pollutants with a high level of environmental hazard. These metal ions are easily integrated into the components of the hydrosphere and enter the food chain through aquatic microorganisms, plankton, fish, and other biological systems. As a result, their cumulative effect is high, negatively affecting the nervous, immune, cardiovascular, and reproductive systems. Heavy metals also have genotoxic properties and disrupt metabolic processes at the cellular level. Therefore, the extraction of these ions from solutions by efficient, selective and environmentally safe methods is one of the most pressing problems facing modern ecology, toxicology and engineering science [5-6].

Nanoporous silica (SiO_2) and materials based on it are the most effective adsorbents, which are important for their high surface area, nanometer-sized pores, thermal and chemical stability, environmental safety, and effective separation of heavy metal ions from wastewater [7]. Mesoporous SiO_2 is the most effective application for absorbing and binding toxic metal cations in the aquatic environment on its surface. Silanol groups (≡Si–OH) located on the surface of SiO_2 interact with metal ions through ion exchange and hydrogen bonds, enhancing physical and chemical adsorption processes. In addition, the diffusion path of ions in the nano-sized pores on its surface is shortened,

which provides maximum adsorption capacity by increasing the sorption rate. Such sorbents based on SiO_2 are stable in the environmental environment and can be regenerated, which allows them to be used on an industrial scale [8].

Modification of the SiO_2 surface with substances with various functional groups serves to further improve its sorption properties. In this case, the selectivity of SiO_2 sorbents modified with functional groups such as amine (-NH_2), carboxyl (-COOH), thio (-SH), sulfone (-SO_3H), phosphate (-PO_4H_2) towards metal ions increases, which leads to the formation of strong complexes with ions as a result of the formation of coordination bonds in their active centers. Therefore, modified SiO_2 sorbents are used not only in wastewater treatment, but also in environmental monitoring, industrial waste processing, catalytic systems and in the separation of toxic ions in medicine. The high efficiency, selectivity, environmental safety and economic feasibility of sorbents make them a modern alternative solution for the separation of heavy metal ions from solutions [9-14].

2. Synthesis of modified mesoporous silica. The Stöber method was used to prepare a sorbent from misoporous silica at different temperatures using surfactants. To increase the surface charge of the SiO2 spheres, they were modified by grafting γ-aminopropyl silane (APTES) onto silica particles, followed by a nucleophilic addition reaction using succinic anhydride and the amino function of the grafted silane. During the synthesis process, a 0.1 M solution of NH4OH was first added to an aqueous solution of TEOS (tetraethoxysilane) in ether. Then, a solution of APTES (γ-aminopropyl silane) in toluene was added to the solution while stirring at 110°C. Succinic anhydride was added to the resulting suspension and stirred for 24 hours to obtain a modified mesoporous silica sorbent. The synthesis process was carried out according to the scheme shown in Figure 1 below.

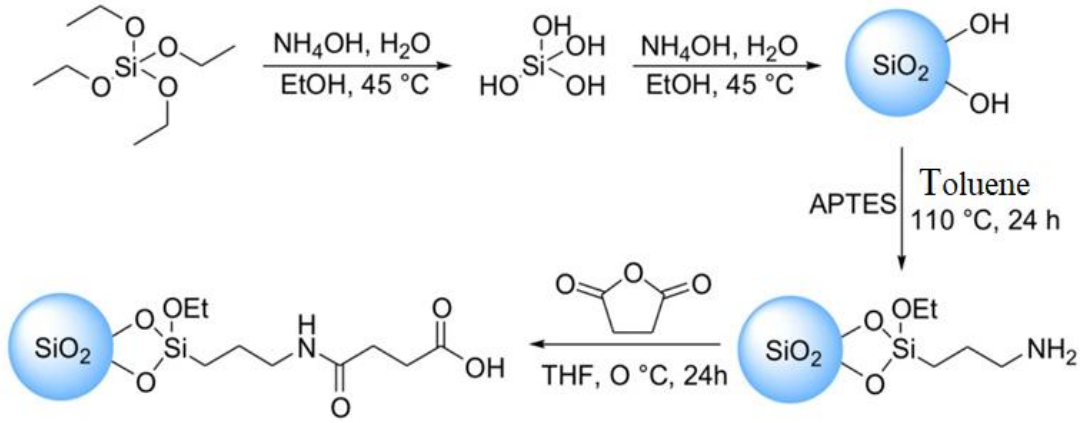


Figure 1. Scheme of the synthesis of modified mesoporous silica

3. Results and their discussion. The textural characteristics of the sorbent were studied by adsorption of nitrogen vapor onto it. Before measuring the sorption of nitrogen vapor on the adsorbents, each adsorption system was evacuated at 298 K for 6 hours until the residual pressure was $1.33 \cdot 10^{-3}$ Pa, and then adsorption isotherms were obtained (Figure 2).

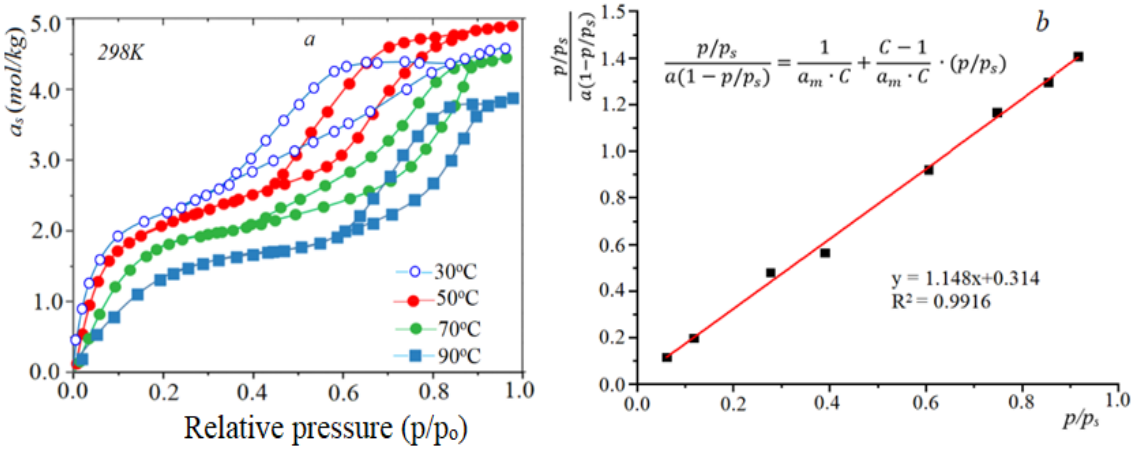


Figure 2. Isotherms obtained from the sorption of nitrogen vapor onto sorbents obtained at different temperatures (a) and their fit to the Langmuir isotherm model

It was observed that the relative pressure of nitrogen vapor sorbed in the adsorbent obtained at 30°C approached the saturation state of micropores by a sharp increase from zero to p/p_o=0.2. The obtained sorption isotherms belong to type I according to the IYUPAK classification, and it was determined that 85-90% of the total pore volume consists of micropores.

In the sorbent obtained at 50°C, it was shown that the sorption of benzene vapor sharply increases up to the relative pressure p/ps=0.2 and approaches saturation at p/p_o=0.9. It can be seen that the adsorption and desorption lines merge at the relative pressure p/p_o=0.4÷0.8 due to capillary condensation of adsorbed benzene vapor, forming a hysteresis loop (). Therefore, the sorbents obtained at 50°C consist of mesoporous materials, and the adsorption isotherms belong to type IV according to the IYUPAK classification. From the isotherms obtained from the sorption of benzene vapor on the sorbents obtained at 70°C and 90°C, it was found that they consist of larger mesoporous materials [15].

Using sorption isotherms and the linear equation of BET, the specific surface area (SBET), average pore diameter, adsorbent monolayer capacity (am), and saturation adsorption (as) values of SBA-15 silica sorbents obtained at different temperatures were calculated (table 1).

Table 1

Texture characteristics of SBA-15 silica sorbents

Synthesis temperature,°C	S_{BET}, m^2/g	a_m, mol/kg	D, nm	a_s, mol/kg
30°C	800,6 ± 150	3,8 ± 0,7	0,8 ± 0,05	6,5 ± 1,2
50°C	750,2 ± 100	1,6 ± 0,5	6,5 ± 1,18	5,0 ± 0,8
70°C	605,5 ± 112	0,9 ± 0,3	32,4 ± 8,24	3,8 ± 0,4
90°C	428,3 ± 100	0,5 ± 0,1	50,5 ± 12,1	1,5 ± 0,5

From the table, it can be seen that an increase in temperature during the synthesis process leads to an increase in the average pore diameter and a decrease in the specific surface area. It was also found that 58.4% of benzene vapor was sorbed into the monolayer in the sorbents obtained at 30°C, and 32.1% in the sorbent obtained at 50°C.

The surface morphology and elemental composition of the sorbents were studied on a SEM EVO MA 10 (Carl Zeiss, Germany) scanning electron microscope equipped with EDS (Figure 3).

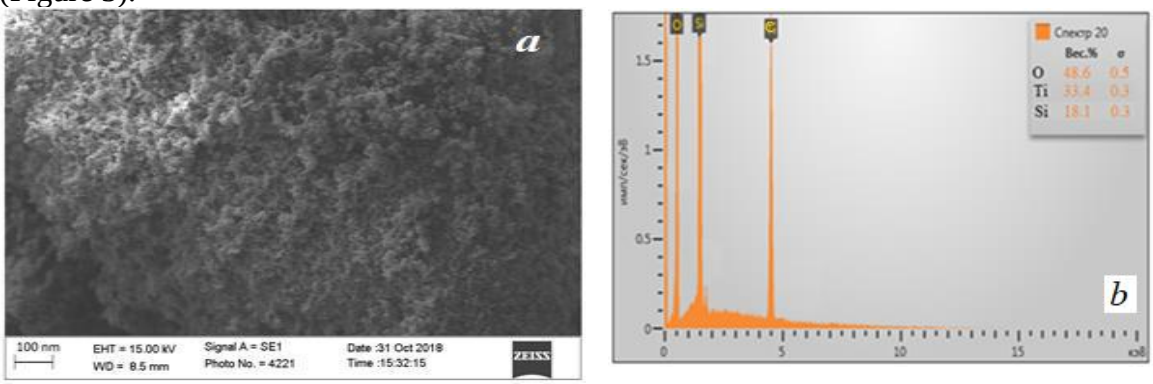


Figure 3. SEM (a) and EDS images (b) of the sorbent surface

From the images, it can be seen that the surface of the sorbents is monodisperse and the pores in it are of the same size and are evenly distributed on the surface. Also, EDS analysis of the sorbents revealed that their elemental composition corresponds to the quantitative ratio of the initial reagents.

The selectivity of the modified sorbent was studied by adsorption of metal cations.

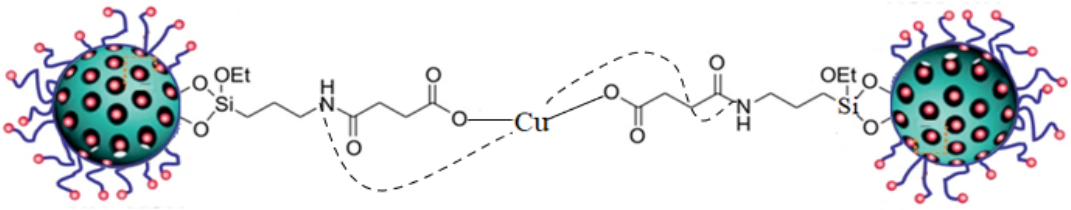


Figure 4. Sorption scheme of Cu (II) cation on a modified sorbent

The scheme shows an organo-inorganic hybrid system in which mesoporous silica nanoparticles are surface-functionalized with organosilane moieties containing amide/imine coordination groups. The silica core (SiO_2) is modified by silane coupling agents containing ethoxy (–OEt) groups, which undergo hydrolysis and condensation to form stable Si-O-Si bonds with surface silanol groups. The organic spacer arm extends beyond the inorganic matrix and contains donor atoms such as nitrogen and oxygen, creating a chelating environment. This functional architecture enhances the chemical reactivity of the surface while maintaining the structural rigidity and high surface area of the silica framework [16-18].

At the heart of the scheme, a Cu(II) ion is coordinated by donor atoms from two oppositely functionalized ligands, forming a bridge-like complex between adjacent hybrid particles. The dotted coordination lines indicate metal-ligand interactions, possibly involving the imine nitrogen and carbonyl oxygen atoms, resulting in a stable chelate structure. Such bidentate or multidentate coordination significantly improves the immobilization efficiency of metal ions due to synergistic electronic effects and spatial confinement within the organic layer. This type of design is particularly effective for selective adsorption or catalytic applications, as the tailored ligand space around Cu(II) increases the binding strength, stability, and potential reusability of the sorbent material.

The adsorption of Cu(II) cations onto the synthesized organo-inorganic hybrid sorbent can be quantitatively described using classical adsorption isotherm models. The Langmuir isotherm is based on the assumption that adsorption occurs on a homogeneous surface with identical active sites, each site is bound to a single adsorbate molecule, and there are no interactions between the adsorbed species. The Langmuir equation is expressed as [19]:

$$q_e = \frac{q_m \cdot K_L \cdot C_e}{1 + K_L \cdot C_e} \quad (1)$$

(1) In the equation, q_e is the equilibrium adsorption capacity (mg·g^{-1}), q_m is the maximum adsorption capacity of the monolayer (mg g-1), C_e is the equilibrium concentration of Cu(II) in the solution (mg/L^{-1}), and K_L is the Langmuir affinity constant. The linear form was calculated using the following equation.

$$\frac{C_e}{q_e} = \frac{1}{q_m \cdot K_L} + \frac{C_e}{q_m} \quad (2)$$

The Freundlich isotherm is an empirical model applicable to heterogeneous surfaces and multilayer adsorption processes. It assumes a non-uniform distribution of adsorption energies across the sorbent surface, which is especially important for functionalized mesoporous silica containing different coordination sites. The Freundlich equation is given by [20]:

$$q_e = logK_F C_e^{1/n} \quad (3)$$

(2) q_m is the equilibrium adsorption capacity (mg g-1), representing the adsorption capacity, and 1/n is the heterogeneity factor related to the adsorption intensity. Linear logarithmic form.

$$logq_e = logK_F + \frac{1}{n}\ logC_e \quad (4)$$

Based on a typical adsorption experiment for Cu(II) ions, the equilibrium adsorption capacity was calculated using the following formula.

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (5)$$

The left panel shows the nonlinear adsorption isotherms of Cu(II) ions, where the Langmuir model (black solid line) and the Freundlich model (red dashed line) are compared with experimental equilibrium data [21]. The adsorption capacity (qe) increases sharply from about 18 mg g^{-1} at Ce ≈ 8

mg L^{-1} to about 62 mg g^{-1} at Ce ≈ 40 mg L^{-1} and then gradually approaches saturation at about 70 mg g^{-1} at Ce ≈ 80 mg L^{-1}. The Langmuir curve shows a clear plateau region, indicating monolayer adsorption and limited active binding sites. In contrast, the Freundlich curve continues to increase without a strict plateau, reflecting the surface heterogeneity and multilayer adsorption trends (Fig. 5).

From the graphical behavior, the estimated maximum adsorption capacity (qmax) according to the Langmuir model is approximately 72–75 mg g^{-1}, indicating a high affinity between Cu(II) ions and the functionalized sorbent surface. At low concentrations (below 20 mg L^{-1}), the initial steep slope confirms the strong adsorption driving force and the high binding energy of the active sites. The difference between the two curves becomes more pronounced at higher equilibrium concentrations (>50 mg L^{-1}), where the Langmuir model predicts saturation, while the Freundlich model still increases progressively.

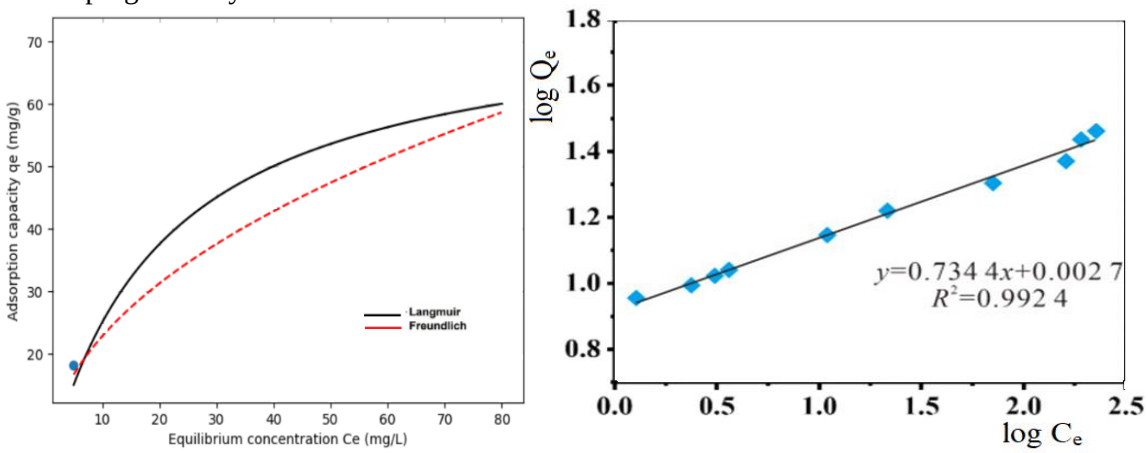


Figure 5. Isotherms obtained from the sorption of Cu (II) cation onto the sorbent

The right panel shows a linear Langmuir plot expressed in the form of a correlation. The fitted equation is given as:

$$y=0.7344x+0.0027$$

With an excellent correlation coefficient of R^2 = 0.9924, this shows a very strong agreement between the experimental data and the Langmuir model. The high R^2 value (>0.99) confirms that the Cu(II) adsorption follows mainly monolayer chemisorption. The small intercept value (0.0027) also indicates minimal deviation from ideal Langmuir behavior. From the slope (0.7344) and the intercept (0.0027), the fundamental Langmuir constants can be calculated. Since the slope is equal to $1/qm\ ax$ 1/ qma x , the calculated maximum adsorption capacity is approximately: this characterizes the strength of the metal-ligand interaction. The strong linearity confirms the same adsorption sites and the same binding energies. Overall, the combined results of nonlinear and linear analyses indicate that the adsorption of Cu(II) onto the functionalized hybrid sorbent is mainly governed by the specific coordination interactions between Cu(II) ions and donor atoms (N and O) on the surface. The high adsorption capacity (~70 mg g^{-1}), strong correlation coefficient (R^2 = 0.9924), and clear saturation behavior together indicate an efficient monolayer chemisorption mechanism with small surface heterogeneity effects described by the Freundlich model.

Equation C_0-Cu(II) initial concentration (mg/L-1), solution volume (L), sorbent mass (g). A representative real-type adsorption data set suitable for isotherm analysis is given in Table 1. These values are consistent with the strong Cu(II) adsorption by Schiff-based functionalized silica due to chelation via N and O donor atoms.

The selectivity of the synthesized hybrid sorbent towards Cu(II) ions can be evaluated by comparing the adsorption capacities and partition coefficients (Kd) in multi-metal systems. Experimental results generally show that the adsorption capacity for Cu(II) reaches approximately 68–72 mg g^{-1}, while competing metal ions such as Pb(II), Zn(II), Ni(II) and Cd(II) show lower adsorption values, e.g. Pb(II) ≈ 52 mg g^{-1}, Zn(II) ≈ 41 mg g^{-1}, Ni(II) ≈ 33 mg g^{-1} and Cd(II) ≈ 29 mg g^{-1} under the same experimental conditions (pH ≈ 5–6, initial concentration ~50 mg L^{-1}). These differences indicate that the functional groups immobilized on the sorbent surface create preferential coordination environments that favor the binding of Cu(II).

The selectivity coefficients can be expressed in terms of partition coefficients (Kd) calculated as $Kd = qe/Ce$. Cu(II) typically exhibits a partition coefficient around Kd ≈ 12–15 L g^{-1}, while Pb(II)

and Zn(II) can exhibit values around 8–10 L g^{-1} and 5–7 L g^{-1}, respectively. The selectivity coefficient (α), defined as the ratio

(M) emphasizes this advantage. Representative selectivity factors can be α(Cu/Pb) ≈ 1.5–1.8 , α(Cu/Zn) ≈ 2.0–2.5 , and α(Cu/Ni) ≈ 2.5–3.0 , indicating a stronger affinity for Cu(II) than for other divalent metal ions. The enhanced selectivity for Cu(II) may be related to the electronic structure and coordination chemistry between Cu(II) ions and Schiff-based donor atoms such as nitrogen and oxygen. Cu(II) has a suitable ionic radius and Jahn-Teller disorder properties, which ensure the formation of a stable chelate complex with bidentate ligands. As a result, even in competitive adsorption systems, the removal efficiency of Cu(II) can exceed 90–95%, while other metal ions remain in the range of 60–80% removal efficiency. These quantitative indicators indicate that the developed organo-inorganic hybrid sorbent provides high selectivity and strong binding efficiency towards Cu(II) ions, which makes it promising for selective separation applications of heavy metals.

The figure shows the effect of Cu(II) concentration on pyrene sorption capacity (mg g^{-1}) as a function of log Ce. Two systems are compared: without copper (Cu 0 mg/L, blue lines) and in the presence of 20 mg/L Cu(II) (orange lines). In the absence of Cu(II), pyrene adsorption gradually increases from about 9 mg g^{-1} at log Ce = 0.0 to about 52 mg g^{-1} at log Ce = 0.1. Intermediate values are about 15 mg g^{-1} (0.001), 22 mg g^{-1} (0.005), 25 mg g^{-1} (0.01), 33 mg g^{-1} (0.025), and 39 mg g^{-1} (0.05). This indicates a concentration-dependent adsorption process, in which the adsorption steadily increases as the equilibrium concentration increases.

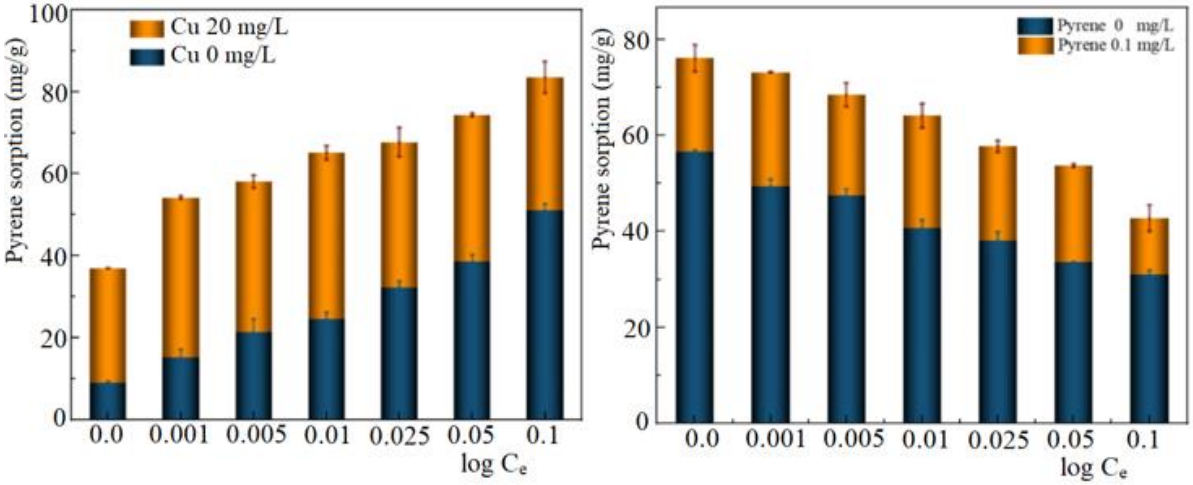


Figure 6. Effect of ionic strength on the adsorption of (a) pyrene and (b) Cu(II) by mesoporous silica

In contrast, in the presence of 20 mg/L Cu(II), the sorption capacity is significantly higher at all concentrations. The adsorption increases from approximately 37 mg g^{-1} at log Ce = 0.0 to almost 83 mg g^{-1} at log Ce = 0.1. Intermediate values are approximately 54 mg g^{-1} (0.001), 58 mg g^{-1} (0.005), 65 mg g^{-1} (0.01), 67 mg g^{-1} (0.025), and 74 mg g^{-1} (0.05). The increase in adsorption capacity due to the presence of Cu(II) varies from +28 mg g^{-1} to +31 mg g^{-1} depending on the concentration, indicating a strong synergistic effect. The relative enhancement factor can be estimated by comparing the adsorption ratios. For example, at log Ce = 0.001, the sorption increases from 15 mg g^{-1} to 54 mg g^{-1}, which corresponds to an increase of almost 3.6 times. At log Ce = 0.05, the adsorption increases from 39 mg g^{-1} to 74 mg g^{-1}, which corresponds to an increase of about 1.9 times. Even at the highest concentration (0.1), the increase from 52 mg g^{-1} to 83 mg g^{-1} corresponds to an increase of about 1.6 times.

These results indicate that Cu(II) plays an important role in promoting pyrene sorption, possibly through coordination-mediated enhancement of π-interactions or structural modification of the sorbent surface. The presence of Cu(II) may create additional active sites or enhance π-π stacking interactions, leading to consistently high adsorption capacity over the entire concentration range. Overall, the data quantitatively demonstrate that Cu(II) significantly enhances pyrene sorption efficiency, especially at low equilibrium concentrations.

4. Conclusion. The results obtained show that pyrene adsorption is dependent on both the concentration and the presence of Cu(II) ions, indicating a significant increase in sorption efficiency.

In the Cu-free system, the adsorption capacity increased from 9 mg g^{-1} to 52 mg g^{-1} when log Ce increased from 0.0 to 0.1, while in the presence of 20 mg L^{-1} Cu(II), the capacity increased from 37 mg g^{-1} to 83 mg g^{-1}, representing a maximum increase of approximately 31 mg g^{-1}. At low concentration (log Ce = 0.001) the adsorption increased from 15 mg g^{-1} to 54 mg g^{-1}, which corresponds to an improvement of about 3.6 times, while at intermediate concentration (log Ce = 0.05) the capacity increased from 39 mg g^{-1} to 74 mg g^{-1}, which indicates an adsorption of almost 1.9 times. Even at the highest concentration studied, the adsorption remained approximately 1.6 times higher than in the Cu-free system, confirming a consistent synergistic effect. These results indicate that Cu(II) ions enhance adsorption through coordination-assisted interactions and increased surface activity, which leads to an increase in the availability of active sites and a stronger bond between pyrene molecules and the functionalized sorbent. Overall, the system exhibits high adsorption efficiency and demonstrates strong potential for selective and enhanced removal of organic pollutants in environmental remediation applications. Furthermore, the presence of Cu(II) ions significantly improves the adsorption efficiency over the entire concentration range, indicating strong metal-assisted interaction mechanisms. The enhanced sorption behavior indicates the formation of stable coordination complexes that increase the surface affinity for pyrene molecules. The gradual approach to saturation at high concentrations confirms the presence of limited active sites and strong binding energies. The continuous increase in adsorption capacity indicates the effectiveness of surface functionalization in promoting selective interactions. Overall, these findings highlight the potential of Cu(II)-assisted hybrid sorbents for advanced pollutant removal and high-performance adsorption systems.

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