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EVALUATION OF THE SORPTION PROPERTIES OF ORGANO/INORGANIC MS-SCHIFF HYBRID SORBENTS TOWARDS METAL CATIONS

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Abstract. Mesoporous silica sorbent modified with Schiff base and free Schiff base was synthesized. The sorbent showed high adsorption efficiency towards Cu(II) ions, which was confirmed by structural (FTIR) and equilibrium studies. FTIR analysis revealed the formation of an azomethine (C=N) at 1630÷1650 cm^{-1} and a subsequent shift of about 10÷15 cm^{-1} from Cu(II) or Pb(II) coordination, indicating strong N,O-bidentate chelation. The adsorption equilibrium data were well described by Langmuir and Freundlich isotherm models, confirming the favorable adsorption behavior. The Langmuir model was found to have a monolayer chemisorption with a maximum adsorption capacity (q_{max}) of about 60÷65 $mg \cdot g^{-1}$ for Cu(II) or Pb(II), reflecting the same active sites formed by the immobilized Schiff groups. The high Langmuir affinity constant (K_L) indicated strong metal-ligand interactions. Meanwhile, the Freundlich model showed good linearity with 1/n<1, confirming the favorable adsorption on a moderately heterogeneous surface and the possibility of multilayer contributions at high concentrations (up to 200 $mg \cdot L^{-1}$).

Keywords: MS-Schiff sorbent, chelate complex, Langmuir and Freundlich isotherm models, FTIR spectroscopy, SBA-15, sorption isotherm.

Organo/noorganik ms-schiff gibrid sorbentning metall kationlariga nisbatan sorbsiya xossalarini baholash

Annotatsiya. Shiff asosi bilan modifikatsiyalangan va erkin Shiff asosli mezog‘ovak kremnezem sorbenti sintez qilindi. Sorbent Cu(II) ionlariga nisbatan yuqori adsorbsiya samaradorligini ko‘rsatdi, bu strukturaviy (FTIR) va muvozanat tadqiqotlari bilan tasdiqlangan. FTIR tahlili 1630÷1650 sm^{-1} da azometin (C=N) hosil bo‘lishini va keyinchalik Cu(II) yoki Pb(II) koordinatsiyasidan taxminan 10÷15 sm^{-1} ga siljishini aniqladi, bu kuchli N,O-bidentat xelatlanishini ko‘rsatadi. Adsorbsiya muvozanati ma'lumotlari Langmuir va Freundlix izoterma modellari tomonidan yaxshi tavsiflangan bo‘lib, bu qulay yutish xatti-harakatlarini tasdiqladi. Langmuir modeli Cu(II) yoki Pb(II) uchun maksimal adsorbsiya sig‘imi (q_{max}) taxminan 60÷65 $mg \cdot g^{-1}$ bo‘lgan monoqatlamli xemosorbtsiyaga egaligi aniqlandi, bu immobilizatsiyalangan Shiff guruhlari tomonidan hosil qilingan bir xil faol joylarni aks ettiradi. Yuqori Langmuir affinlik konstantasi (K_L) kuchli metall-ligand o‘zaro ta'sirini ko‘rsatdi. Shu bilan birga, Freundlich modeli 1/n<1 bilan yaxshi chiziqlilikni ko‘rsatdi, bu o‘rtacha geterogen sirtda qulay adsorbsiyani va yuqori konsentratsiyalarda (200 $mg \cdot L^{-1}$ gacha) ko‘p qatlamli hissa qo‘shish mumkinligini tasdiqladi. Freundlich konstantasi (K_F) sorbentning yuqori adsorbsiya intensivligini yanada qo‘llab-quvvatladi. Langmuir

va Freundlich modellari o‘rtasidagi izchillik shuni ko‘rsatadiki, adsorbsiya asosan sirt heterojenligi effektlari bilan birga keladigan monoqatlamli xemosorbsiya orqali sodir bo‘ladi.

Kalit so‘zlar: MS-Schiff sorbent, xilat kompleks, Langmuir va Freundlix izoterma modellari, FTIR spectroscopy, SBA-15, sorbsiya izotermasi.

Оценка сорбционных свойств органо-неорганического гибридного MS-Schiff сорбента по отношению к катионам металлов

Аннотация. Был синтезирован мезопористый кремнезёмный сорбент, модифицированный основанием Шиффа, а также сорбент на основе свободного основания Шиффа. Полученный сорбент продемонстрировал высокую адсорбционную эффективность по отношению к ионам Cu(II), что подтверждено структурным анализом (FTIR) и исследованиями равновесия. Анализ FTIR выявил образование азометиновой группы (C=N) в области 1630–1650 $см^{-1}$ и её последующее смещение примерно на 10–15 $см^{-1}$ при координации Cu(II) или Pb(II), что указывает на сильное N,O-бидентатное хелатирование.

Данные адсорбционного равновесия хорошо описываются моделями изотерм Лэнгмюра и Фрейндлиха, что подтверждает благоприятный характер процесса сорбции. Согласно модели Лэнгмюра, установлено наличие монослойной хемосорбции с максимальной адсорбционной ёмкостью (q_{max}) для Cu(II) или Pb(II) порядка 60–65 $мг \cdot г^{-1}$, что отражает однородные активные центры, сформированные иммобилизованными группами основания Шиффа. Высокая константа сродства Лэнгмюра (K_L) свидетельствует о сильном взаимодействии металл–лиганд.

В то же время модель Фрейндлиха демонстрирует хорошую линейность при $1/n < 1$, что подтверждает благоприятную адсорбцию на умеренно гетерогенной поверхности и возможность вклада многослойной сорбции при высоких концентрациях (до 200 $мг \cdot л^{-1}$). Константа Фрейндлиха (K_F) дополнительно подтверждает высокую интенсивность адсорбции сорбента. Согласованность моделей Лэнгмюра и Фрейндлиха указывает на то, что процесс адсорбции в основном протекает по механизму монослойной хемосорбции с участием эффектов поверхностной гетерогенности.

Ключевые слова: MS-Schiff сорбент, хелатный комплекс, модели изотерм Лэнгмюра и Фрейндлиха, FTIR-спектроскопия, SBA-15, изотерма сорбции.

1. Introduction. In the last two decades, the rapid growth of industry and production, urbanization and intensive use of metal-containing materials have led to a significant increase in heavy metal pollution in natural waters and industrial waste [1]. Heavy metal cations such as Cu^{2+}, Pb^{2+}, Ni^{2+}, Cd^{2+} and Hg^{2+} are non-biodegradable and tend to accumulate in living organisms, posing a serious threat to the health of ecosystems such as humans, animals and plants. Today, traditional treatment methods often have low selectivity, limited efficiency at low concentrations and high economic costs [2-3]. Therefore, the development of highly efficient, selective and renewable sorbent materials for the removal of metal ions from aqueous environments is becoming one of the most pressing problems in modern environmental and materials chemistry.

Mesoporous silica (SiO_2) has attracted the attention of researchers as a promising application for sorbent design due to its high specific surface area, ordered pore structure, thermal stability, and chemical inertness [4-5]. Mesoporous silica sorbents such as MCM-41 and SBA-15 are uniquely designed nanomaterials that adsorb heavy metal cations and volatile organic compounds. However, the natural surface of SiO_2 mainly contains silanol (Si-OH) groups, which show limited affinity for metal cations. This limitation requires targeted surface modification to introduce functional groups capable of strong and selective coordination with metal ions [6].

Modification of the mesoporous SiO_2 surface with organic materials containing various functional groups leads to the formation of organo/inorganic hybrid sorbents with improved structural advantages of inorganic matrices and high chemical reactivity of organic ligands. By introducing functional groups such as amino (–NH_2), thiol (–SH), carboxyl (–COOH), and Schiff base (–C=N–) onto the mesoporous SiO_2 surface, it is possible to create special coordination centers that serve as chelating sites for metal cations. Such hybrid systems increase the adsorption capacity, selectivity, and tunable sorption properties, which in turn allows for the effective separation of toxic metal cations from wastewater [7].

The evaluation of the sorption properties of these hybrid materials requires a comprehensive study of adsorption isotherms, kinetics, pH effect, surface charge behavior, and regeneration ability [8-10]. Models such as Langmuir and Freundlich isotherms, together with pseudo-first-order and pseudo-second-order kinetic models, provide fundamental insights into the adsorption mechanism and the nature of the interaction between the sorbent surface and metal ions. Understanding these physicochemical parameters is crucial for optimizing the design and practical performance of the sorbent [11-13]. Also, the practical applications of organic/inorganic hybrid sorbents cover many areas, such as wastewater and drinking water treatment, hydrometallurgy, analytical preconcentration of trace metals, and environmental monitoring. Their high stability, reusability, and flexibility make them suitable for industrial-scale applications. As a result, the modification of mesoporous SiO_2 surfaces and the evaluation of their sorption properties toward metal cations represent a very relevant and influential area of modern research in materials and environmental chemistry [14].

2. Synthesis of modified mesoporous silica.

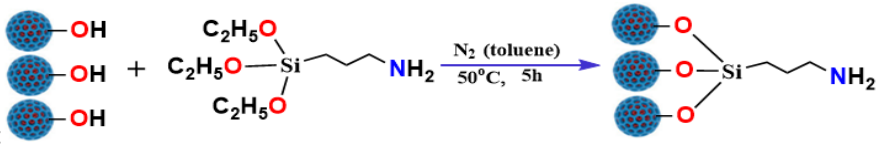
2.1. Materials and methods. In the synthesis of mesoporous sorbent, APTES Jinan Xinggao Chemical Technology Co., Ltd, China was used as the SiO_2 source. Purity > 98,6 %), ethanol as the solvent (purity > 94,6 %), 0.01 M HCl solution as the hydrolysis catalyst and to control the pH of the solution, cetyltrimethylammonium chloride (triblock copolymer-P123, Jinan Xinggao Chemical Technology Co., Ltd, China. Purity > 99,2 %). Salicylic aldiged (purity > 96,5%) and aniline (purity > 98,4%) were used to form Schiff bases.

2.2. **Synthesis of modified mesoporous silica**

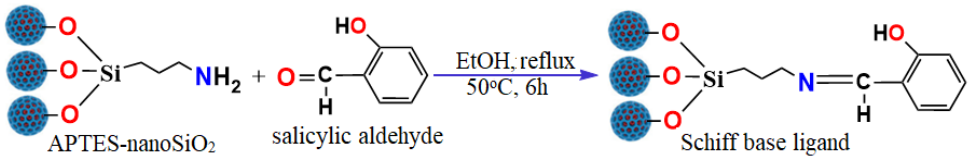
The synthesis was carried out using sol-gel technology. The sol-gel technology is used to obtain nanomaterials in a simple reactor. The method is notable for its simplicity of equipment, economy, environmental safety, and low cost of the resulting product. It is also characterized by the mild conditions of the hydrolytic polycondensation reaction of the initial precursors and the possibility of introducing functional group monomers, polymers, and oxides of variable valence metals into the reaction system, the use of a single solvent for all reagents, and the ability to control the structure and size of the final product. In the sol-gel process, organic/inorganic components can be mixed in any ratio and at the nanoscale.

The synthesis of a mesoporous silica sorbent containing a Schiff base ligand was carried out at 50°C in an acidic environment. The synthesis process is as follows:

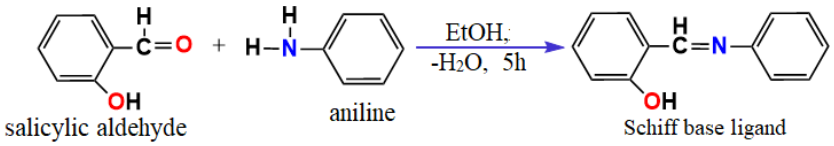
1. Mesoporous nanosilica was formed by attaching APTES to mesoporous silica initially prepared from TEOS. The reaction was carried out at 50°C for 6 hours in a nitrogen atmosphere:



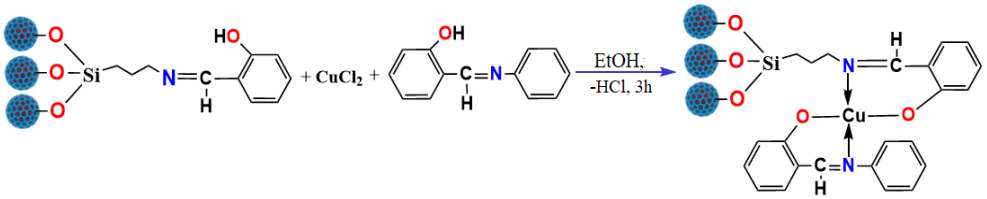
2. An organo/inorganic hybrid nanocomposite of mesoporous silica consisting of a Schiff-based ligand was synthesized by attaching salicyl aldehyde to the resulting APTES-silica nanocomposite in a toluene environment at 50°C:



3. A Schiff base ligand was synthesized from salicyl aldehyde and aniline in ethanol medium:



4.The sorption of Cu (II) cations from aqueous solutions using an organo/inorganic hybrid nanocomposite of mesoporous silica and a free Schiff base ligand was investigated through the following reaction scheme:



When $CuCl_2$ is introduced into the reaction system, copper(II) ions form coordination bonds with the ligand bound to the mesoporous silica surface and the donor atoms of the free Schiff base ligand. The formation of these coordination bonds results in the formation of a stable hybrid organo-inorganic chelate complex. The adsorption properties of heavy metal ions such as Cu(II) and Pb(II) are mainly directly related to the functionalization of the sorbent surface. The MS-Schiff sorbent has a rigid, high-surface-area mesoporous silica framework functionalized with covalently attached Schiff base moieties-usually formed by the condensation of APTES (3-aminopropyltriethoxysilane) with salicylaldehyde. This results in pendant –C=N– (imine) and –OH, $–NH_2$ donor groups distributed within the porous network. In contrast, an unbound (free) Schiff ligand sorbent lacks this supporting silica architecture and acts primarily as a small molecule chelating agent with limited surface area and disordered accessibility [15].

3. Results and their discussion.

3.1. FTIR spectroscopy. To confirm the gradual formation of the Schiff base ligand immobilized on the surface and the subsequent formation of coordination bonds with Cu(II) ions, its FTIR (Fourier transform infrared spectroscopy) spectra were studied (Fig. 1). The red spectrum in the figure is the spectrum of the azomethine (Schiff base) modified with APTES and subsequently immobilized with salicylaldehyde. The appearance of the absorption band around 1630÷1650 cm^{-1} of the spectrum is associated with the C=N stretching vibration, confirming the formation of an imine bond in the sorbent. The broad band around 3400 cm-1 of the spectrum corresponds to the O-H and residual silanol groups in the phenol ring, and the peaks at 2920÷2550 cm^{-1} correspond to the aliphatic $–CH_2–$ groups of the propyl chain derived from APTES. The peaks at about 1100 cm^{-1} indicate the presence of Si–O–Si siloxane bonds of the silicon framework, and the peaks around 1250÷1300 cm^{-1} are associated with the valence vibrations of the C–O bonds in the phenol[16]. The black spectrum is the spectrum of the coordination complex formed by the interaction of the surface-bound Schiff base, an additional free Schiff base ligand in solution, and the Cu (II) cation. In the region of ~1640 cm-1 of the spectrum, a significant shift to lower regions (~1600÷1610 cm^{-1}) is observed due to the vibrations of the C=N bonds, which indicates that the azomethine nitrogen forms coordination bonds with the copper ion. Similarly, the shift to lower regions of the peaks formed by the valence vibrations of the C–O bonds in the phenol confirms the formation of a bond between the oxygen atom in the phenol and the Cu (II) cation. These spectral changes provide strong evidence for the chelation of Cu(II) by the Schiff base ligand through both nitrogen and oxygen donor atoms [17].

In addition, new weak absorption peaks appearing in the spectrum in the region of 500÷600 cm^{-1} are associated with Cu-N and Cu-O coordination bonds that are absent in the red spectrum. The

signal corresponding to Si-O-Si bonds around 1100 cm^{-1} remains unchanged in both spectra, indicating that the silicon structure retains its structural integrity during the complexation process. Overall, the FTIR analysis results confirm the immobilization of the Schiff base on the silicon surface and the formation of a stable N_2O_2-type Cu(II) coordination complex within the hybrid organo-inorganic material.

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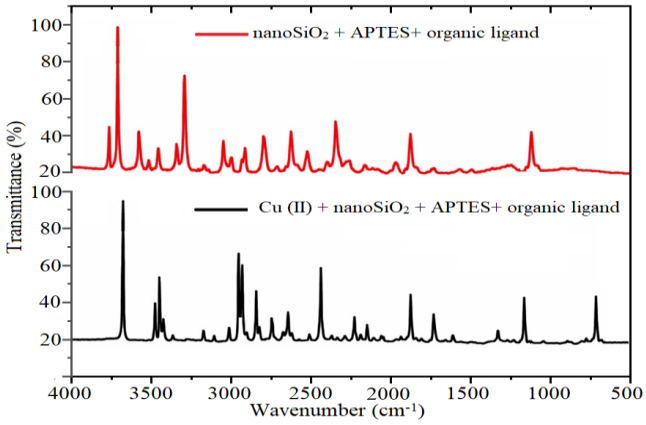


Figure 1. FTIR spectrum of the chelate complex formed from Cu(II) adsorbed on MS-Schiff sorbent

The signal corresponding to Si-O-Si bonds around 1100 cm^{-1} remains unchanged in both spectra, indicating that the silicon structure retains its structural integrity during the complexation process. Overall, the FTIR analysis results confirm the immobilization of the Schiff base on the silicon surface and the formation of a stable N_2O_2-type Cu(II) coordination complex within the hybrid organo-inorganic material. To study the textural characteristics and sorption properties of the modified mesoporous silica, an isotherm was obtained from the sorption of nitrogen vapors at 298 K (Fig. 2*a*). The obtained sorption isotherm was found to belong to the type IV H2 type according to the IYUPAK classification [18-20]. From the figure, it can be seen that the sorption isotherm sharply increases to the relative pressure p/p_o=0.2, and the saturation of the micropores in the range p/p_o=0.2÷0.9 - mesopores. The fact that the adsorption and desorption lines merge at the relative pressure p/p_o=0.5÷0.8 due to capillary condensation of the adsorbed nitrogen vapors, forming hysteresis loops, indicates that the main part of the total pores in the sorbents consists of mesopores.

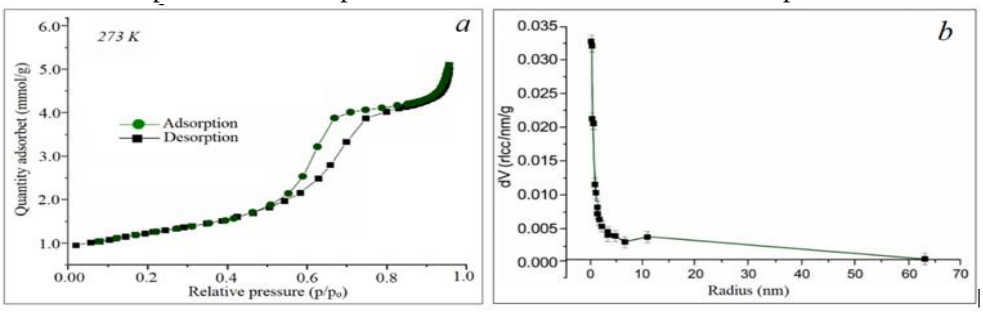


Figure 2. Isotherms obtained from nitrogen adsorption onto MS-Schiff sorbent-Cu(II) complex

The pore size distribution plot obtained from the nitrogen adsorption data allows us to compare the mesoporous silica material before modification with the sample functionalized with the Schiff-based ligand formed by APTES and salicyl aldehyde (Fig. 2*b*). The sharp increase in dV(r) values in the region of small radii (≈2÷6 nm) in the graph indicates that the material has a typical mesoporous structure, which is characteristic of the SiO_2 matrix obtained by sol–gel technology. After modification, a relative decrease in dV(r) is observed in this mesoporous range, which indicates that the internal volume of the pore is partially filled as a result of the chemical bonding of APTES molecules to the pore walls through Si–O–Si bonds with silanol groups and subsequent condensation

with salicyl aldehyde to form a bulky Schiff ligand. Nevertheless, the fact that the maximum of the distribution is maintained precisely in the mesoporous region indicates that the structural integrity of the silicon matrix is not compromised and that the modification process has only chemically altered the internal surface, without destroying the pore architecture.

The low and flat appearance of *dV(r)* values towards large radii (≈10÷60 nm) indicates the presence of macropores or interparticle spaces, but after modification, no significant change is observed in this area. This confirms that the Schiff ligand is mainly located on the mesoporous walls and does not affect the external interparticle space. As a result, along with the high degree of mesoporosity preserved in the sorbent, the inner surface is chemically enriched with donor atoms (–N and –O), which indicates the appearance of active centers favorable for the subsequent formation of coordination complexes with metal ions. Thus, the distribution of pores based on nitrogen adsorption scientifically substantiates not only the mesoporous nature of the structure, but also the successful modification process, i.e., the formation of a functional surface while preserving the structure.

Using the results obtained from the studied systems, the important textural characteristics of the sorbents were calculated using the BET equation: specific surface area (S_{BET}, $m^2 \cdot g^{-1}$), pore volume (V_s, $cm^3 \cdot g^{-1}$), average diameter (D, nm), and nitrogen vapor sorption capacities (as, mol/kg) (Table 1).

Table 1

Texture characteristics of MS-Schiff sorbent for nitrogen bond adsorption

Modified sorbent	S_{BET}, $m^2 \cdot g^{-1}$	V, $cm^3 \cdot g^{-1}$	D, nm	a_s, mol/kg	ρ, g/cm^3
nanoSiO_2 Schiff basis	350 ± 45,3	0,82 ± 0,05	5,4 ± 0,8	3,52±0,6	0,45±0,02

3.2. **Adsorption kinetics** Kinetic analysis of adsorption rate is a common method to study the mechanism of adsorption process control, which helps to optimize the processing conditions of adsorbents. The study of the kinetic properties of metal cation sorption onto an orgono/inorganic hybrid sorbent usually requires an appropriate model. In this study, two simple models, including pseudo-first-order and pseudo-second-order kinetic models, were used and compared. The linear form of both models can be given as the following equations (1) and (2) [21]:

$$\log(Q_e - Q_t) = -\frac{k_1}{2.303} + logQ_e \quad (1)$$

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{t}{Q_e} \quad (2)$$

In the equation, Q_e ($mg \cdot g^{-1}$) and Q_t ($mg \cdot g^{-1}$) are the amounts of metal ions adsorbed at equilibrium and at a given adsorption time t (min), respectively, where k_1 (min^{-1}) is the rate constant of the pseudo-first-order model, and k_2 ($g \cdot mg^{-1} \cdot min^{-1}$) is the rate constant of the pseudo-second-order model.

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Table 2

First- and second-order kinetics of metal cation sorption onto MS-Schiff sorbent

Metal ions	Pseudo-first order		Pseudo- second order		
	k_1 (min^{-1})	R^2	k_2 ($g \cdot mg^{-1} min^{-1}$)	$Q_{e(cal)}$ ($g \cdot mg^{-1}$)	R^2
Cu (II)	0.0128	0.918	0.0052	45.6	0.977
Pb (II)	0.0152	0.912	0.0095	38.4	0.928

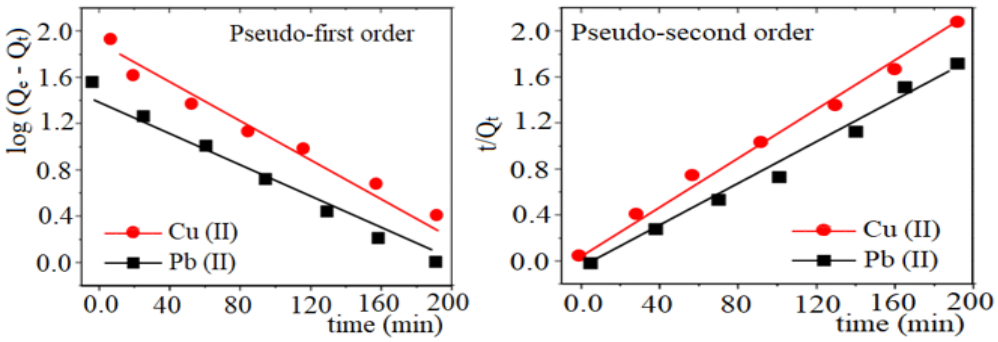


Figure 3. Pseudo first-and second-order kinetic curves

In addition, it was found that the adsorption of metal ions onto the sorbent was very rapid, and that the chemical adsorption process, through electron exchange, allows the rate of sorption to be controlled.

3.2. Adsorption isotherm. The sorption properties of the MS-Schiff sorbent were evaluated by isotherms obtained by sorption of Cu(II) and Pb(II) cations onto it (Fig.4). The sorption isotherms revealed a strong interaction between the sorbent and the metal ions. According to the Langmuir model, the maximum monolayer adsorption capacity (Q_{max}) for Cu(II) reached approximately 62 mg·g^{-1}, and for Pb(II) at high equilibrium concentrations (~180÷200 mg L^{-1}) it reached approximately 50 mg·g^{-1}. The rapid initial increase in the adsorption capacity below 50 mg L^{-1} indicates a high density of accessible coordination sites corresponding to imine (–C=N–) and phenolic oxygen donor groups located in the mesoporous silica framework. The stronger Langmuir lines observed for Cu(II) indicate a higher binding affinity (K_L) than for Pb(II), consistent with the tendency of Cu(II) to complex more strongly to nitrogen and oxygen donor atoms in Schiff-based ligands [20].

Freundlich isotherms support favorable adsorption behavior over the entire concentration range. The Freundlich curves show a continuous increase without a strict saturation, reaching almost 95 mg g^{-1} for Cu(II) and 88 mg g^{-1} for Pb(II) at 200 mg·L^{-1}, indicating contributions from heterogeneous surface interactions and multilayer adsorption. The steeper Freundlich curves for Cu(II) indicate a higher KF constant and a stronger adsorption intensity (1/n < 1), which confirms energetically more favorable binding sites for copper ions. In general, a quantitative comparison of the Langmuir and Freundlich models shows that the adsorption on the MS-Schiff sorbent is mainly monolayer chemisorption with moderate surface heterogeneity, and the Cu(II) cation exhibits approximately 20÷25% higher adsorption capacity than the Pb(II) cation, which indicates the selectivity of the Schiff functionalized mesoporous silica towards copper ions.

The adsorption mechanism was studied by analyzing the isotherms using the Langmuir and Freundlich models. The fit of the Langmuir and Freundlich isotherm models to the linear forms (3) and (4) was examined [22-24]:

$$\frac{C_e}{Q_e} = \frac{C_e}{Q_m} + \frac{1}{Q_M \cdot K_L} \quad (3)$$

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

In the equation, Ce (mg L−1) is the equilibrium concentration of Pb(II) and Cu(II) ions in the solution, Qe (mg L−1) defines the ions in the equilibrium state on the adsorbents, Qm (mg· g^{-1}) is the theoretical maximum adsorption capacity, KL (L, mg^{-1}) is the Langmuir isotherm constant depending on the adsorption energy, K_F (mg·g^{-1}) is the Freundlich parameter, and n is the Freundlich heterogeneity coefficient depending on the nature of the adsorption. The Langmuir model is generally suitable for modeling monolayer adsorption on uniform surface areas, and the Freundlich model is often used for non-ideal multilayer adsorption that occurs on heterogeneous surface areas.

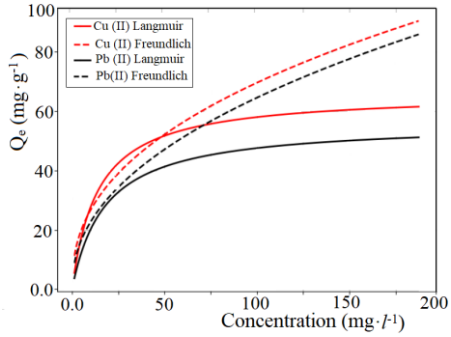


Figure 4. Langmuir and Freundlich isotherms obtained from the sorption of metal cations onto MS-Schiff sorbent

Figure 6 shows the Langmuir and Freundlich isotherm plots for the adsorption of Pb(II) and Cu(II) onto MS-Schiff sorbent. The fitted isotherm parameters and linear regression correlation coefficients are given in Table 3. It is observed that the Freundlich model has a lower R^2 correlation coefficient, while the Langmuir model has a value close to 1. The results show that the Langmuir model is more suitable for fitting the experimental data. The fitting of the Langmuir isotherms shows that the adsorption sites are uniformly distributed on the surface of the adsorbents and the adsorption is carried out by the formation of monolayers through a complexation mechanism.

Table 3

Physical quantities of metal cation adsorption on MS-Schiff sorbent

Metal cations	Langmuir model			Freundlich model		
	K_L (L, mg^{-1})	$Q_{m(kal)}$ (mg·g^{-1})	R^2	1/n	K_F (mg·g^{-1})	R^2
Cu (II)	0.0964	62,4	0.973	0.370	10.4	0,979
Pb (II)	0.207	49,5	0.992	0.207	9.5	0,912

The adsorption process may be limited by the diffusion of metal ions within the porous channels. To study the adsorption rate of metal ions, the amount of Cu(II) and Pb(II) ions adsorbed on the complex was plotted against the interaction time. It was observed that the adsorption rate of Cu(II) and Pb(II) on the organo/inorganic adsorbent was very fast during the first 10 min of adsorption. After 60 min of interaction, the removal of the two ions on the sorbent increased slowly and the adsorption index was found to reach a maximum.

The linear Langmuir plots (C_e/q_e and C_e) for Cu(II) and Pb(II) adsorption onto the MS-Schiff sorbent were observed to increase over the studied concentration range (up to ~200 mg g^{-1}), confirming that the adsorption process is predominantly monolayer and occurs at energetically identical active sites (fig 5). The high correlation and almost parallel trends indicate the strong surface homogeneity provided by the covalently immobilized Schiff-base functional groups within the mesoporous silica framework. The steep slope observed for Pb(II) compared to Cu(II) indicates a relatively lower maximum adsorption capacity (q_{max}) for Pb(II), while Cu(II) exhibits a higher affinity constant (K_L), reflecting stronger coordination interactions with the imine nitrogen and phenolic oxygen donor atoms.

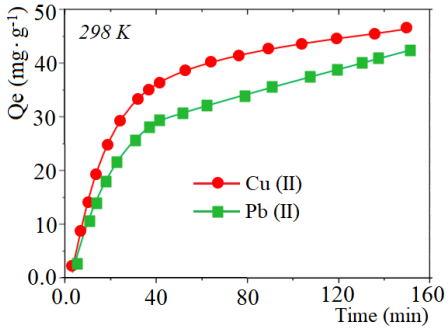


Figure 5. Effect of contact time on adsorption performance of Pb(II) and Cu(II) on MS-Schiff sorbent

These results confirm that chemisorption through specific metal-ligand complexation dominates the absorption mechanism in the MS-Schiff system. Freundlich line plots (log q_e and log C_e or C_e/q_e plot depending on the linearity) were also found to be steep, indicating that adsorption occurs on a moderately heterogeneous surface with energetically distributed binding sites. The higher intercept and slope of Cu(II) compared to Pb(II) indicate a higher Freundlich constant (KF) and a stronger adsorption intensity (1/n<1), indicating more favorable binding of copper ions. The fit of the data to the Freundlich model implies that in addition to monolayer chemisorption, secondary interactions such as surface heterogeneity and layered contributions within the mesopores at high concentrations may also be involved. Overall, the combined Langmuir and Freundlich analyses demonstrate that the MS-Schiff sorbent provides high affinity, moderate surface heterogeneity mainly towards Pb(II), and clear selectivity towards Cu(II) [25].

The adsorption selectivity of the MS-Schiff sorbent towards Cu(II) and Pb(II) ions can be justified based on the principles of coordination chemistry, surface functionality, and physicochemical properties of the metal ions. The Schiff base functional groups immobilized on the mesoporous silica surface contain imine nitrogen (–C=N–) and phenolic oxygen donor atoms that act as border Lewis bases. According to Pearson theory, Cu(II), classified as a border acid with a relatively high charge density and a smaller hydrated ionic radius (~4.19 Å), forms stronger and more directional coordination bonds with nitrogen and oxygen-containing ligands than Pb(II), which is relatively softer and has a larger hydrated radius (~4.01÷4.50 Å, depending on the hydration state). In addition, Cu(II) benefits from a ligand field stabilization energy (LFSE), which increases the thermodynamic stability of Cu-Schiff complexes. These factors together lead to high affinity constants and high adsorption capacity for Cu(II), as reflected in the isotherm parameters.

In addition to the intrinsic metal-ligand affinity, structural aspects of the MS-Schiff sorbent significantly affect selectivity. The uniform distribution of Schiff-based groups within the mesoporous channels provides well-defined chelation sites capable of forming stable five- or six-membered coordination rings with Cu(II) and promotes inner-sphere complexation. Pb(II), due to its larger ionic size and stereochemically active lone pair, often forms more distorted coordination geometries, which can reduce optimal coverage with ligand donor atoms within the confined mesopores. Diffusion effects within the nanoscale channels further favor Cu(II), as its smaller effective ionic size facilitates faster transport and access to internal binding sites.

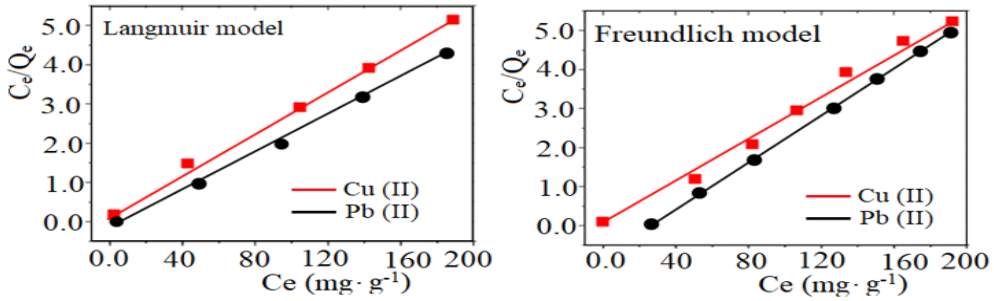


Figure 6. Compatibility of the Langmuir and Freundlich models for the sorption of Cu(II) and Pb(II) ions onto MS-Schiff sorbent

As a result, the adsorption selectivity arises from a combination of thermodynamic stability of Cu-ligand complexes, geometric compatibility within the mesoporous structures, and kinetic advantages, leading to consistently higher absorption and stronger binding of Cu(II) relative to Pb(II).

Conclusion. Adsorption studies clearly demonstrate that the MS-Schiff sorbent exhibits high efficiency and clear selectivity towards Cu(II) and Pb(II) ions, with a consistently high selectivity for Cu(II). Langmuir and Freundlich isotherm analyses confirm that the adsorption occurs mainly via monolayer chemisorption accompanied by moderate surface heterogeneity. The higher maximum adsorption capacity and affinity constants obtained for Cu(II) indicate stronger coordination interactions between copper ions and the imine nitrogen and phenolic oxygen donor atoms of the immobilized Schiff-base ligand. These findings confirm that functionalization of mesoporous silica with APTES-salicylaldehyde-derived Schiff groups significantly enhances metal binding performances compared to unstructured or free ligand systems.

In general, the selectivity mechanism is driven by a synergistic combination of the thermodynamic stability of the Cu-ligand complexes, the favorable geometric fit within the mesoporous channels, and the diffusion-controlled accessibility of the active sites. The structural integrity of the mesoporous silica structure ensures a uniform distribution of chelating sites, which provides efficient and predictable adsorption behavior. Therefore, the MS-Schiff sorbent can be considered a very promising material for the selective removal of Cu(II) and Pb(II) from aqueous media, especially in applications requiring high affinity, stability, and controllable adsorption performance.

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