Removal of molybdenum from wastewater using modified amino-functional framework: A study of the adsorption properties

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Abstract
Herein, we have employed a convenient method to study kinetic and thermodynamic parameters of the adsorption of molybdenum from wastewater by using modified Zr-based metal–organic frameworks, UIO-66-NH2-X, as adsorbents. The frameworks were fabricated with a simple and fast method from terephthalic acids and Zirconium metal ions and modified via introduction of aldehyde groups using imine condensation. This is the first report in which MOFs have been utilized as adsorbents in molybdenum adsorption. The prepared adsorbents show high adsorption efficiency, which is representative of an improvement compared to other literature. The adsorption capacity and molybdenum removal for the modified framework are 667 mg/g and 93%, respectively which is a significant and remarkable value among other previously reported articles. The experimental adsorption data could be well interpreted with the pseudo-second-order kinetics and the Langmuir isotherm model. Thermodynamic studies illustrated a spontaneous exothermic mechanism for the adsorption process.

KEYWORDS
high adsorption capacity, metal organic framework, molybdenum adsorption, post-synthetic modification, superior adsorbent

1 | INTRODUCTION

Molybdenum is one of the active transition elements with the biological application as well as used in industry. It is a vital nutrient for both humans and plants. In plants, it is indispensable to fix atmospheric nitrogen and convert nitrate into nitrite, while in humans it is needed for removing toxins and activating enzymes. On the other hand, from industrial viewpoint, molybdenum has miscellaneous functions such as vacuum tubes, alloying agents, rubber, catalysts, and solid lubricants. However, the excess effluent concentration of molybdenum in water caused crucial environmental problems. Therefore the removal of molybdenum from wastewater is essential in order to eliminate its hazardous impact on the environment.

Different oxidation states of Molybdenum vary from Mo(II) to Mo(VI) exist in wastewater which among them molybdate ion (MoO₄²⁻) is the most soluble species. One of the most significant radionuclides is Molybdenum-99 (⁹⁹Mo) which is used in nuclear medicine. ⁹⁹Mo is one of the toxic compounds in radioactive waste that need to be removed in order to the protection of people and the environment.

Various methods have been investigated for molybdenum removal from wastewater such as chemical precipitation, adsorption process, ion exchange, electrochemical techniques, and biological treatment. So far, among diverse available wastewater treatments, adsorption process is significant due to its effectiveness, easy separation, and suitable economical condition. In general, developing environmentally friendly adsorbents with high adsorption
capacity toward molybdenum adsorption from wastewater is still a major challenge among scientists. Many different adsorbents have been developed and applied in order to eliminate Mo(VI) from wastewater such as carbon nanotubes, pyrite, ferric oxide gel, chitosan, ion exchange resin, and so forth.\textsuperscript{18–20}

Metal–organic frameworks (MOFs) are a new class of porous materials that have attracted significant attention of many researchers in recent years due to their unique and special characteristics including high surface area and porosity, tunable pore structure, high thermal stability, and different accessible sites.\textsuperscript{21} These compounds are formed via the auto-assembly of metal ion clusters and polyfunctional organic linkers. Up to now, they have widely employed in numerous applications such as catalysis, sensing, adsorption, sequestration, drug delivery, and so forth.\textsuperscript{22–26} In particular, they have great potential to be as an adsorbent for heavy metal wastewater treatment. Although MOFs are applied to adsorb various metals from wastewater, no research has been reported on molybdenum adsorption by MOFs so far. Furthermore, MOFs are much more efficient in adsorbing heavy metals compared to other adsorbents owing to their unique characteristics.

Based on the above consideration, herein, we attempted to design and synthesize a novel and new framework as an efficacious adsorbent for the adsorption of Mo(VI) from wastewater. To the best of our knowledge, there is no report on the improvement of molybdenum removal from wastewater using heterogeneous adsorbents based on MOFs, and the reported adsorbents in this work have been designed for the first time for effective molybdenum adsorption.

Therefore, the adsorption studies on molybdenum pollutant from wastewater was investigated over modified MOFs. With this regard, UIO-66-NH$_2$ has been successfully synthesized and modified with aldehyde functional groups using imine condensation (Scheme S1). The adsorption capacity, kinetics, and thermodynamic have been evaluated in detail. Additionally, the equilibrium data were analyzed by using Langmuir isotherms. The details of the adsorption process and its effective factors will be discussed.

## 2 | EXPERIMENTAL SECTION

### 2.1 | Materials and instrumentation

Materials and instrumentation are explained in supporting information.

### 2.2 | Synthesis of adsorbents

Synthesis of adsorbents are demonstrated in supporting information.

## 3 | RESULTS AND DISCUSSION

In the present work, UIO-66-NH$_2$ was synthesized and successfully modified with aldehyde groups using imine condensation reaction (Scheme S1) in order to make a superior adsorbent for removal of molybdenum from wastewater.

### 3.1 | Characterization of adsorbents

The FT-IR spectra of the prepared compounds are depicted in Figure S1. The FT-IR spectrum of UIO-66-NH$_2$ (Figure S1a) exhibits characteristic bands at 1388 and 1641 cm$^{-1}$ which is attributed to the symmetric and asymmetric stretching vibrations of the carboxylic acid functional group, respectively. Additionally, the bands appeared around 719 and 761 cm$^{-1}$ can be associated with the out-of-plane deformation vibration of C–H groups in the phenyl rings of 2-aminoterephthalic acid linkers.\textsuperscript{27} In the FT-IR spectra of UIO-66-NH$_2$-SA and UIO-66-NH$_2$-OH (Figure S1b,c), the absence of the relevant band for the C=O stretching vibration at 1640 cm$^{-1}$ is owing to the overlapping with the asymmetric COO stretching vibrations.

The crystalline nature of the prepared structures was investigated by powder x-ray diffraction (PXRD) analysis and the results are represented in Figure S2. Based on the comparison of the main diffraction peaks of the as-synthesized and simulated structures, the structural accommodation of compounds can be concluded and no apparent loss of crystallinity was observed. Furthermore, it can be observed that topology and crystallinity of the structures have retained upon modification of the framework.\textsuperscript{28}

To specify the morphology and particle size of synthesized compounds, scanning electron microscopy (SEM) was conducted and the micrographs are presented in Figure S3. The SEM images indicate uniform particle size of about 100 nm for UIO-66-NH$_2$ (Figure S3a,b). In addition, there is no substantial changes in the morphology of the structures after modification of the framework (Figure S3c,d).

To determine the specific surface area, porosity, and the pore volume of the prepared frameworks, $N_2$ adsorption–desorption analysis at low temperatures was assessed and the results are summarized in Figure 1 and Table S1. The $N_2$ adsorption–desorption isotherms exhibited type I isotherms for both pristine MOF and modified frameworks. According to the values of textural parameters in Table S1, the surface area and pore volume for UIO-66-NH$_2$ were 886 m$^2$/g and 0.45 cm$^3$/g, respectively, whereas a substantial decrease for the corresponding values was observed owing to the modification of the framework. Therefore, the surface area and pore volume for UIO-
66-NH2-SA and UIO-66-NH2-OH decreased to 574 m²/g and 0.31 cm³/g, and 436 m²/g and 0.25 cm³/g, respectively which affirmed the successful modification of the framework.

3.2 | Adsorption study of the synthesized frameworks

To evaluate the adsorption proficiency of the prepared frameworks, the adsorption of Mo was studied using UIO-66-NH2-SA and UIO-66-NH2-OH as the adsorbents. The pristine MOF (UIO-66-NH2) have no capability for Mo adsorption. Therefore, the adsorption process was only performed with modified frameworks. Additionally, the effect of contact time, temperature, pH, pollutant concentration, and the amount of adsorbents was also investigated in the adsorption process.

3.3 | Molybdenum removal ability of the synthesized compounds

The removal of molybdenum from wastewater (Mo. R.) was calculated by the following equation:

\[
\text{Mo.R. (\%)} = \left( \frac{C_0 - C_t}{C_0} \right) \times 100,
\]

where \(C_0\) and \(C_t\) are the initial and the final concentration at contact time \(t\) (mg/L), respectively.

The results of the adsorption capacity and the amount of removed Mo are summarized in Table 1. According to the results, high Mo removal and adsorption capacity were observed for both adsorbents due to the modification of the framework. However, the adsorption performance of UIO-66-NH2-SA is slightly better than UIO-66-NH2-OH. On the other hand, the results demonstrated that the pristine MOF (UIO-66-NH2) does not the capability to adsorb molybdenum by itself and therefore, cannot remove it from wastewater. However, modification of the framework caused a remarkable enhancement in molybdenum removal and adsorption capacity from 6 to 93% and 13–667 mg/g, respectively. The effect of the modification will be discussed later. Hence, the better performance can be observed via the modification of the framework.

3.4 | Effect of different parameters on adsorption of molybdenum

3.4.1 | The influence of contact time on the molybdenum adsorption

The effect of contact time on Mo adsorption was investigated using UIO-66-NH2-SA and UIO-66-NH2-OH at room temperature and the results are presented in Figure S5. Based on the results, in Figure S5, it can be observed that Mo adsorption was increased with the increase of the reaction time for both adsorbents. However, the amount of molybdenum adsorption remained constant after a certain time which referred to as the equilibrium time for the reactions. Therefore, the equilibrium time for UIO-66-NH2-SA and UIO-66-NH2-OH were obtained 210 and 900 min, respectively.

3.4.2 | The effect of pH on adsorption of molybdenum

The solution pH is one of the most significant factors during the adsorption process which can affect the active site charge of the
adsorbent, the ionization degree, and consequently the structure of the adsorbate molecules. Thus, the solution pH was evaluated in the range of 3–11. The pH of the solution was adjusted by 0.1 M HCl or 0.1 M NaOH, and the results are depicted in Figure S6. As can be observed in Figure S6, with the enhancement of the solution pH from 3 to 7, the amount of removed Mo increased, and then decreased upon the further increment of the solution pH. Thus, the best results for the pH value for both adsorbents were 7.

3.4.3 | The effect of adsorbent dosage

The adsorption process was performed with various adsorbent dosages and the results are summarized in Table S2. The results indicated that Mo removal percentage was enhanced upon increasing of the amount of adsorbent from 10 to 100 mg. Accordingly, 100 mg was obtained as the optimal value for both adsorbents.

3.4.4 | Effect of temperature on adsorption process

In order to evaluate the effect of temperature, the adsorption process was studied at different temperatures in the presence of UIO-66-NH₂-SA and UIO-66-NH₂-OH as adsorbents (Table S3). According to the results, the amount of removed Mo reduced upon temperature increment from 298 to 318 K. Thus, room temperature is the favorable temperature for the adsorption process. Adsorption isotherms with respect to the temperature are discussed later.

3.4.5 | The effect of molybdenum concentration

To elucidate the effect of pollutant, adsorption experiments were conducted with the diverse initial concentrations of Mo using UIO-66-NH₂-SA and UIO-66-NH₂-OH as adsorbents and the results are presented in Table S4. Based on the results, it is obvious that Mo removal decreased upon increasing Mo concentration owing to the occupation of active sites of the adsorbents.

3.4.6 | Adsorption kinetics

The study of adsorption kinetics is one of the significant factors in the adsorption process as it provides noteworthy information about the diffusion mechanism and the reaction pathway as well as the effect of time in the removal of pollutants. For this purpose, two common adsorption kinetic models were utilized including pseudo-first-order and pseudo-second-order equations, which are illustrated completely in the supporting information.

The adsorption kinetics of UIO-66-NH₂-SA and UIO-66-NH₂-OH were investigated through monitoring the concentration of Mo at various contact times and the results are exhibited in Figures S7 and S8. Additionally, the kinetic adsorption parameters and the correlation coefficient values ($R^2$) calculated from the fitting line are shown in Table S5. Based on the results, the higher $R^2$ values for the pseudo-second-order model in comparison to the pseudo-first-order model demonstrated that the results were fitted well with the pseudo-second-order kinetics model which expressed that the adsorption process proceeds via chemisorption mechanism.

3.4.7 | Adsorption isotherms

Adsorption isotherms specify the interaction between adsorbate molecules and the active site on the adsorbent surface. Among several presented isotherm models, two Langmuir and Freundlich's models have been frequently used for investigation of the experimental data of adsorption isotherms which are explained in the supporting information.

Figures S9 and S10 demonstrate the results of Langmuir and Freundlich's models of Mo adsorption over UIO-66-NH₂-SA and UIO-66-NH₂-OH as adsorbents at 298 K in the initial concentration range of Mo from 20 to 200 mg/L. Moreover, the calculated parameters for both Langmuir and Freundlich's models in the presence of adsorbents are listed in Table S6. According to the results, since the Langmuir isotherm shows a better fit with the adsorption experimental data for both adsorbent, the adsorption process occurs uniformly and in a monolayer form.

3.4.8 | Adsorption thermodynamics

To investigate the desirability of the adsorption process, adsorption thermodynamics was studied. The calculation method for thermodynamic parameters has been explained in supporting information.

To evaluate adsorption thermodynamic parameters, adsorption experiments were carried out over UIO-66-NH₂-SA and UIO-66-NH₂-OH as adsorbents at 298 K and the results are depicted in Table S7. Negative values were obtained for $\Delta G$ which revealed that the adsorption process occurred spontaneously.

3.4.9 | The plausible mechanism of the adsorption process

As mentioned above, aldehyde functional groups incorporated into amine groups using imine condensation to form salicylaldehyde imine and hydroxynaphthaldehyde imine (Scheme 1, pathway I). Molybdenum metal has the ability to combine with suitable ligands to increase its coordination number and form octahedral geometry. On the other hand, oxygen and nitrogen atoms have high affinity to interact with metal centers. Therefore, in the next step, after the addition of molybdate anions, molybdenum atoms interacts and forms a Schiff base complex with the oxygen atoms of the hydroxyl groups and the nitrogen atom C=N bonds (Scheme 1, pathway II).
3.4.10 | Comparison adsorption efficiency of the synthesized compounds

According to the results of Table 1, Mo adsorption capacity for UIO-66-NH2-SA and UIO-66-NH2-OH was obtained 666 and 500 mg g\(^{-1}\), respectively. The high adsorption capacity is mainly owing to the accessibility of the oxygen and nitrogen atoms on the formed imines for coordination with molybdate anions. The higher amount of adsorption capacity for UIO-66-NH2-SA than UIO-66-NH2-OH is related to the less steric hindrance of salicylaldehyde compared to 2-hydroxynaphthaldehyde functional groups.

Overall, we were able to achieve excellent results compared to others in very mild condition (room temperature and neutral pH).

In order to investigate the amount of molybdenum leakage absorbed from the prepared adsorbents, the leaching process was studied for adsorbed molybdenum during 2 months. According to the results, the leaching amount of adsorbed molybdenum from adsorbents after a period of 2 months was very low and was reported to be equal to 0.7 and 1.1% for UIO-66-NH2-SA and UIO-66-NH2-OH, respectively.

To assess the proficiency of the prepared adsorbents in view of the recent progress, a comparison was made between our prepared adsorbents and other adsorbents in which diverse compounds were applied to adsorb molybdate anions and the results are summarized in Table 2. In our work, it can be observed that very high adsorption capacity was obtained compared to other reported literature, which is representative of the high activity and efficiency of the prepared adsorbents. The use of very mild conditions (room temperature and neutral pH) to achieve the best results, is another advantage of our work compared to others.

Given that the present molybdenum in industrial effluents (\(^{99}\)Mo) has the same structure with the molybdenum used in this work, the prepared adsorbents can be used to adsorb radioactive molybdenum from industrial wastewater.

### Table 2: The comparison of adsorptive activities with some previously reported adsorbent

| Entry | Adsorbent                        | Adsorbate | Adsorption capacity (mg/g) | Reference |
|-------|----------------------------------|-----------|---------------------------|-----------|
| 1     | Chitosan gel bead                | MO(VI)    | 265                       | [32]      |
| 2     | HFO-PsAX                         | MO(VI)    | 212                       | [33]      |
| 3     | ZHD50                            | MO(VI)    | 18                        | [34]      |
| 4     | DWTRs                            | MO(VI)    | 44                        | [1]       |
| 5     | Goethite                         | MO(VI)    | 26                        | [35]      |
| 6     | ZnFe\(_2\)O\(_4\) nanospheres   | MO(VI)    | 31                        | [36]      |
| 7     | Mesoporous \(\gamma\)-Al\(_2\)O\(_3\) nanospheres | MO(VI) | 56 | [37] |
| 8     | MWCNT(\(\text{NaOCl}\))         | MO(VI)    | 23                        | [38]      |
| 9     | Zeolite-supported magnetite      | MO(VI)    | 19                        | [39]      |
| 10    | Charcoal                         | MO(VI)    | 51                        | [20]      |
| 11    | Carbonized ZnCl\(_2\)           | MO(VI)    | 19                        | [40]      |
| 12    | Zeolite-supported magnetite      | MO(VI)    | 18                        | [41]      |
| 13    | Fe(III)/Cr(III) hydroxide       | MO(VI)    | 12                        | [42]      |
| 14    | UIO-66-NH2-SA                    | MO(VI)    | 667                       | Present work |

### Scheme 1: The plausible mechanism for molybdenum adsorption

In order to investigate the proficiency of the synthesized adsorbent, the recyclability of UIO-66-NH2-SA was carried out and evaluated through successive adsorption experiments under the optimized reaction conditions and the results are displayed in Figure S11. After each cycle, the desired adsorbent was separated and dispersed in a mixture of 2 M HCl and 1 M HNO\(_3\) in order to remove the adsorbed...
molybdenum, washed with ethanol, dried at 100°C, and then utilized in the next subsequent adsorption experiment. According to the results, the prepared absorbent can adsorb molybdenum with high molybdenum removal percentage for five sequential runs without loss of its adsorption efficacy. The PXRD patterns of UIO-66-NH₂-SA after five recycle runs confirmed the maintenance of the crystalline structure of the absorbent (Figure S12).

4 | CONCLUSION

In summary, a metal-organic framework with amine functional groups was synthesized and successfully modified with aldehyde functional groups using imine condensation. The prepared materials were utilized as an efficacious absorbent to remove molybdenum from wastewater. This is the first report in which MOFs have been applied as adsorbents in molybdenum adsorption. High adsorption capacity was observed for both adsorbents compared with other previously reported materials. The experimental adsorption data could be well interpreted with the pseudo-second-order kinetics and the Langmuir isotherm model. Thermodynamic parameters indicated an exothermic spontaneous mechanism for the molybdenum adsorption process. Since the present molybdenum in industrial effluents (⁹⁹Mo) has the same structure with the molybdenum used in this work, the prepared adsorbents can be used to adsorb radioactive molybdenum from industrial wastewater.

AUTHOR CONTRIBUTIONS

Mahdie Saghian: Conceptualization (equal); data curation (equal); investigation (equal); validation (equal); writing – original draft (equal).

Saeed Dehghanpour: Data curation (equal); project administration (equal); resources (equal); supervision (equal); writing – review and editing (equal).

Massoomeh Sharbatdaran: Methodology (equal); resources (equal); supervision (equal); writing – review and editing (equal).

DATA AVAILABILITY STATEMENT

Data available on request from the authors.

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SUPPORTING INFORMATION
Additional supporting information can be found online in the Supporting Information section at the end of this article.

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