


Rocks as a sustainable resource mSiO₂: Extraction, characterisation, and use for heavy metals removal from wastewater by adsorption process

Sofiane Hamid Hussein¹, Wahran M. Saod ^{2*}

¹Department of Chemistry, Faculty of Education for Pure Sciences, University of Anbar

²Department of Chemistry, Faculty of Sciences, University of Anbar

ARTICLE INFO

Received: 31/05/2025
Accepted: 11/08/2025
Available online: 13/03/2026
April Issue
[10.37652/juaps.2025.160965.1403](https://doi.org/10.37652/juaps.2025.160965.1403)

 CITE @ JUAPS

ABSTRACT

Wastewater from various sources can release large amounts of chemical pollutants and related substances into the environment. There is an immediate need for methods to purge water and wastewater of contaminants; one possible strategy is to employ matrix-stabilized nanoparticles for adsorption-based pollutant removal. Mesoporous silica (mSiO₂) can be derived from natural sources as a cost-effective alternative to industrial products. Natural minerals such as porcelainite, kaolinite, shale, and diatomite contain silicon dioxide (SiO₂), making them promising raw materials for the manufacture of mesoporous silica. Mesoporous silica is characterized by a highly ordered pore structure, with diameters ranging from 2 to 50 nm, an excellent surface area, and a slimming structure. Recent studies have demonstrated the adsorption capacity of mSiO₂, particularly modified varieties, for removing toxic metal ions such as lead (Pb²⁺), cadmium (Cd²⁺), and chromium (Cr³⁺), even at low concentrations. These ions pose significant environmental risks due to their bioaccumulative nature and persistence in aquatic environments. Therefore, in simple terms, adsorption is a cost-effective, simple, and efficient method that is comparable to other techniques such as reverse osmosis, membrane filtration, precipitation, oxidation-reduction, and ion exchange.

The mesoporous silica (mSiO₂) was characterized using FTIR, FESEM, and XRD analyses. Fourier transform infrared (FTIR) spectroscopy has been widely used to characterize the functional properties of both tattoo chemicals and the distinctive features of military compounds. A strong short-duration stretching band at 1000 cm⁻¹, attributed to silicon-oxygen vibrations, is the bandgap location of silicon dioxide. This demonstrates the fineness of the prepared material. The XRD pattern of pure silica (mSiO₂) showed a strong stretch at $2\theta = 2.2^\circ$ (100), spanning two stretches at $2\theta = 3.7^\circ$ (110) and 4.3° (200). These lines indicate the hexagonal mesoporous structure of mSiO₂. FESEM images showed that mSiO₂ has a uniform surface composed of densely packed spherical particles.

The test materials were evaluated for the removal of Pb²⁺, Cd²⁺, and Cr³⁺ ions, with removal efficiencies of 97.8% for Pb²⁺, 98% for Cd²⁺, and 99.6% for Cr³⁺ ions. Manufactured silica effectively decomposes water under various conditions, with optimal elimination achieved at pH 8, a contact time of 60 minutes, and a temperature of 298.

Corresponding author

Wahran Saod
sc.wahran.s@uoanbar.edu.iq

Keywords: Mesoporous silica (mSiO₂), Porcelains rock, Silica Extraction, Waste water ions (Pb²⁺, Cd²⁺, Cr³⁺)

1 INTRODUCTION

In response to the growing global demand for eco-friendly nanomaterials, researchers have increasingly

turned to extracting mesoporous silica (mSiO₂) from natural sources as sustainable and cost-effective alternatives to industrial products [1]. Natural rocks such

as porcelanite, kaolinitic minerals, volcanic tuff, and diatomite are rich in silicon dioxide (SiO_2), making them promising raw materials for the synthesis of mesoporous silica [2]. Economically, the use of abundant, low-cost sources such as fly ash, porcelanite, and kaolin enhances the viability of mSiO_2 production, supporting circular-economy strategies and local resource utilization in environmental applications [3]. Mesoporous silica is distinguished by its high surface area, customizable pore architecture, and organized porous structure, with pore diameters ranging from 2 to 50 nm [4]. Additionally, its surface chemistry can be readily modified with organic or inorganic functional groups, making it highly adaptable for a wide range of environmental applications [5], especially in adsorption-based water purification systems.

Recent research demonstrated that mSiO_2 , particularly its modified forms, has high adsorption effectiveness in eliminating harmful heavy metal ions such as hexavalent chromium (Cr^{3+}), cadmium (Cd^{2+}), and lead (Pb^{2+}) [6], even at low concentrations. The environmental and health risks posed by these ions are substantial due to their bioaccumulative nature and persistence in aquatic environments [7]. Adsorption, therefore, stands out as a simpler, more affordable, and more effective treatment approach than other methods such as ion exchange, chemical precipitation, membrane filtration, reverse osmosis, and oxidation–reduction [8]. Improving the adsorption capacity of mesoporous silica requires surface modification. Functional components such as amines and thiols ($-\text{SH}$) have demonstrated selectivity toward specific metal ions, significantly improving uptake performance, with some reports showing capacities exceeding 200 mg g^{-1} for Pb^{2+} ions [9, 10]. The adsorption mechanisms typically involve physical adsorption, ion exchange, and surface complexation and are influenced by parameters such as solution pH, temperature, and surface functionalization [11]. Moreover, the structural and chemical stability of mesoporous silica allows repeated use through simple regeneration processes, rendering it suitable for long-term industrial deployment. The use of adsorption isotherm models such as Langmuir and Freundlich further aids in understanding the nature and capacity of the adsorption process, providing insights into whether interactions are monolayer or multilayer and whether they are homogeneous or heterogeneous [12].

The increasing discharge of heavy metals from industries such as battery manufacturing, tanning, mining, refining, and the production of pesticides, paints, and dyes has become a significant source of aquatic con-

tamination [13, 14]. In addition, natural phenomena such as volcanic activity, spring water leaching, erosion, and microbial processes contribute to elevated concentrations of these metals in water bodies [15]. While trace amounts of heavy metals are essential for certain biological functions, excessive accumulation can lead to severe health effects, including carcinogenicity, nephrotoxicity, and neurological damage [16]. Against this backdrop, the development of high-performance and environmentally friendly adsorbents has become a research priority. Among the promising candidates, mesoporous silica derived from natural sources stands out for its ease of functionalization, low cost, and distinctive physicochemical characteristics. Therefore, the current study aims to examine the production of mesoporous silica from readily available naturally occurring rocks in the area and to assess its ability to remove toxic metal ions from aqueous solutions. The research also explores the operational parameters affecting adsorption, underlying mechanisms, and theoretical adsorption models, with the goal of establishing a sustainable approach for heavy metal remediation [17]. This study aimed to compare the adsorption behavior of both types of silica under identical experimental conditions, thereby assessing their efficiency in eliminating hazardous metal ions from water-based solutions.

2 MATERIALS AND METHODS

2.1 Materials used in the chemical and natural synthesis of mesoporous silica (mSiO_2)

The synthesis of mesoporous silica (mSiO_2) involves high-purity reagents to ensure the quality of the final product. The primary chemical precursors include tetraethyl orthosilicate (TEOS, $\text{C}_8\text{H}_{20}\text{O}_4\text{Si}$) with a purity of 99% (Evonik Industries; Wacker Chemie, Germany), which serves as the silicon source, and cetyltrimethylammonium bromide (CTAB, $\text{C}_{16}\text{H}_{33}\text{N}(\text{CH}_3)_3\text{Br}$), also with a purity of 99% (Sigma-Aldrich, USA; Alfa Aesar, USA), which acts as the structure-directing agent. Aqueous ammonia (NH_3 , 25%) was used to catalyze the hydrolysis and condensation reactions. For the natural synthesis route, raw materials were derived from natural porcelanite rocks collected from the Department of Geology, College of Science, University of Anbar, Al-Qaim, Iraq. Additional chemicals used in the purification and extraction process include sodium hydroxide (NaOH , 98%; Sigma-Aldrich, USA), sulfuric acid (H_2SO_4 , 97%; Sigma-Aldrich, USA), and hydrochloric acid (HCl , 99%; Sigma-Aldrich, USA).

Lead nitrate ($\text{Pb}(\text{NO}_3)_2$), cadmium nitrate tetrahydrate ($\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$), and chromium nitrate ($\text{Cr}(\text{NO}_3)_3$) were obtained from BDH, UK.

2.2 Preparation of mesoporous silica (mSiO_2) from natural porcelanite rock

The natural porcelanite rock was initially thoroughly washed with distilled water to remove any surface contaminants. The cleaned material was subsequently dried overnight at 90°C to eliminate moisture, followed by manual grinding and sieving through a $180\ \mu\text{m}$ mesh. The processed powder was thermally activated at 500°C for 3 hours to enhance its reactivity. For silica extraction, 2.5 g of the activated sample was combined with 150 mL of 3 M sodium hydroxide (NaOH) in a 250 mL round-bottom flask equipped with a reflux condenser. This mixture was stirred at 300 rpm for 24 h to convert the silica content into soluble sodium silicate. The resulting solution was filtered using ash-free filter paper (Whatman No. 41) and rinsed with hot distilled water. Silica precipitation began by reducing the pH of the clear filtrate to below 10, after which it was gradually neutralized with 5 M sulfuric acid (H_2SO_4) under vigorous stirring until the pH reached 7. This step produced a soft, white silica hydrogel that was allowed to age at room temperature for a full day. After filtration, the gel was washed with distilled water to remove residual sulfate ions. Subsequent drying steps were performed at 80°C and 110°C , each for 24 h. Finally, the dried product was calcined at 800°C for 2 h, yielding a fine, white mesoporous silica powder suitable for environmental applications [18, 19].

2.3 Adsorption of Cd^{2+} , Pb^{2+} , and Cr^{3+} ions

A batch adsorption study was conducted to identify the adsorption capacity of mSiO_2 to adsorb Cd^{2+} , Pb^{2+} , and Cr^{3+} ions at varying amounts (10, 20, 30, 40, and $50\ \text{mg L}^{-1}$), with pH adjustment ($\text{pH} = 8$) and a contact duration of 2 h at a temperature of 298 K. The experiments were conducted using 50 mL centrifuge tubes. Solutions with varying concentrations of Pb^{2+} , Cd^{2+} , and Cr^{3+} were added to the tubes, each containing 10 mL of solution. The samples underwent centrifugation for 10 min at 5,000 rpm to remove any remaining suspended material. The samples were first agitated in a water bath at 100 rpm to ensure optimal interaction between the adsorbent and adsorbate. The percentage removal effectiveness ($\%R$) and the equilibrium adsorption capacity (q_e , mg g^{-1}) were calculated using the corresponding Equations (1) and (2) [20].

2.4 Batch adsorption assessment of heavy metals using mesoporous silica (mSiO_2)

A controlled-batch adsorption procedure was employed to investigate the adsorption efficiency of mesoporous silica (mSiO_2) for Pb^{2+} , Cd^{2+} , and Cr^{3+} ions from aqueous solutions. Different starting metal concentrations (10, 20, 30, 40, and $50\ \text{mg L}^{-1}$) were prepared, and each solution's pH was set to 8. Adsorption was performed over a 2 h contact period at 298 K. For each trial, 10 mL of the metal solution was added to a 50 mL centrifuge tube. The amount of contaminant added refers to the initial concentration of the target pollutant (Pb^{2+} , Cd^{2+} , Cr^{3+}) that was introduced into $10\ \text{mg L}^{-1}$ the solution at the beginning of the experiment. This is a key parameter in adsorption studies, as it influences both the driving force for mass transfer and the adsorption capacity of the material being tested. To ensure adequate contact between the sorbent and the metal ions, the samples were first agitated at 100 rpm in a water bath. To remove any leftover particulate matter, centrifugation was performed for 10 min at 5,000 rpm after agitation [21]. The adsorption performance was quantified by calculating both the equilibrium adsorption capacity (q_e , mg g^{-1}) and the removal efficiency ($\%R$) using standard analytical expressions based on concentration differences before and after treatment [22].

2.5 Adsorption process of heavy metals

Lead (Pb^{2+}) solutions with concentrations of 10, 20, 30, 40, and $50\ \text{mg L}^{-1}$ were prepared in 10 mL for removal experiments. Activated carbon is commonly used as an adsorbent. Its weights (0.01, 0.02, 0.03, 0.04, and 0.05 g) were added to each Pb^{2+} solution. All samples were incubated in a shaking incubator at 25°C for 60 min, agitated at 150 rpm, except those used to assess the impact of contact time. The time-dependent samples were processed for 15-180 min. The equilibrium adsorption capacity (q_e) and removal efficiency ($\%R$) were calculated using Equations 1 and 2, respectively. The pH, temperature, time, and starting concentration of heavy metal ions determined the carbon quantity, which varied from 10 to 50 mg [22, 23].

$$\%R = \frac{c_0 - c_e}{c_0} \times 100 \quad (1)$$

$$q_e = \frac{(c_0 - c_e) \times V(L)}{m(\text{mg})} \quad (2)$$

The variables in this equation include the initial

concentration C_0 , which represents the starting metal ion concentration (mg/L). C_e : is the equilibrium metal ion concentration (mg g⁻¹). V is the solution's volume (L). m : is the adsorbent's mass (g).

$$\frac{C_e}{q_e} = \frac{1}{K_L \times q_m} + \frac{C_e}{q_m} \quad (3)$$

where K_L and q_m are the Langmuir constants.

$$\ln q_e = \ln K_F + \left(\frac{1}{n}\right) C_e \quad (4)$$

where K_F and n are the Freundlich constant

3 RESULTS AND DISCUSSION

3.1 Infrared spectroscopy using fourier transforms

Fourier transform infrared (FTIR) spectroscopy is widely employed to identify functional groups in both pure substances and composite materials and to compare structural features between compounds. The technique relies on the vibrational motions of atoms and molecules, which correspond to specific infrared absorption frequencies. The FTIR spectrum of mesoporous silica (mSiO₂), as illustrated in Figure 1, exhibits prominent absorption bands in the 1400–1700 cm⁻¹ and 800–1000 cm⁻¹ regions. A particularly strong peak appears near 1000 cm⁻¹ [24,25], which is attributed to Si–O stretching vibrations typically associated with silicate materials and mesoporous silica frameworks. Additionally, all analyzed samples display a characteristic band at approximately 790 cm⁻¹, corresponding to the symmetric stretching of Si–O–Si linkages and indicative of the silica network structure, as shown in Figure 1. The appearance of an absorption band in the 2000–2500 cm⁻¹ region in the FTIR spectrum of mesoporous silica is often attributed to the absorption of CO₂ gas from the air during the measurement and its return to the gas itself, or to the presence of residual organic impurities from directing agents (such as CTAB) or organic precursors (such as TEOS).

3.2 Diffraction of x-rays

Patterns of X-rays for mSiO₂ are displayed in Figure 2, which shows the X-ray diffraction (XRD) pattern of pure silica (mSiO₂). The pattern shows a strong peak at $2\theta = 2.2^\circ$ (100) and two relatively weak lines at $2\theta = 3.7^\circ$ (110) and 4.3° (200). These lines indicate the hexagonal mesostructure of mSiO₂ [26].

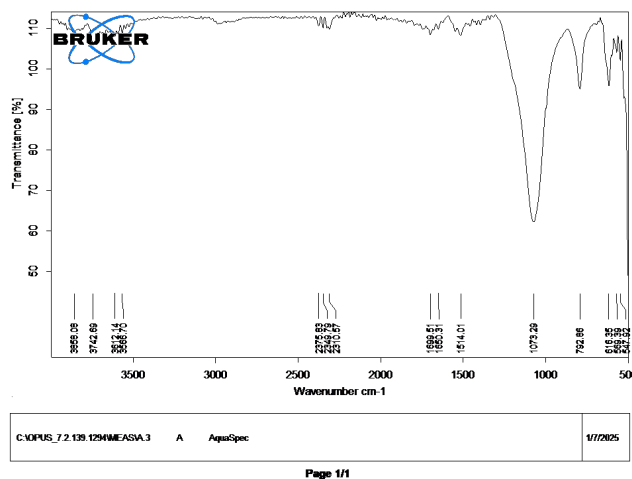


Fig. 1 FTIR spectra of (mSiO₂) by natural source

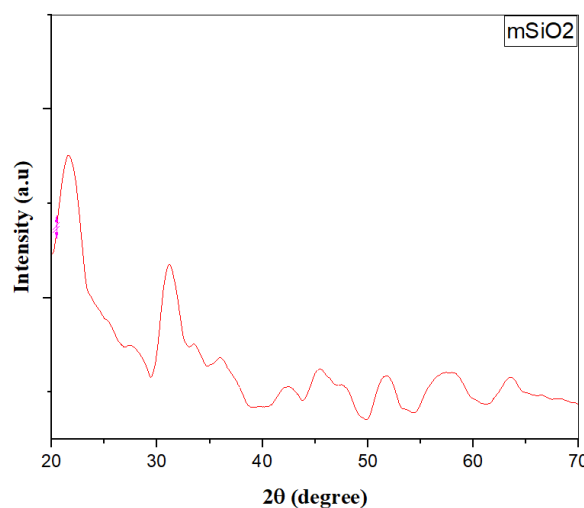


Fig. 2 X-ray diffraction of (mSiO₂) by natural source

3.3 Field emission scanning electron microscopy (FE-SEM) analysis of mesoporous silica(mSiO₂)

To examine surface morphology and microstructural characteristics, field emission scanning electron microscopy (FESEM) was employed, as depicted in Figure 3. The FESEM images of the synthesized material reveal that mSiO₂ exhibits a uniform surface composed of densely agglomerated, quasi-spherical particles. As shown in the micrographs, the particles are very fine, with measured diameters of 42, 48, 51, and 53 nm, indicating that the extracted material consists of particles below 100 nm. The particles appear closely packed, and individual grains are difficult to distinguish due to agglomeration, a

common phenomenon in nanosized silica powders. This morphology is typical of mesoporous silica materials prepared under controlled conditions and is consistent with previously reported structural characteristics of mSiO₂-type materials. The observed particle aggregation may be attributed to the high surface energy of these fine particles, which promotes clustering during the drying and calcination stages [27].

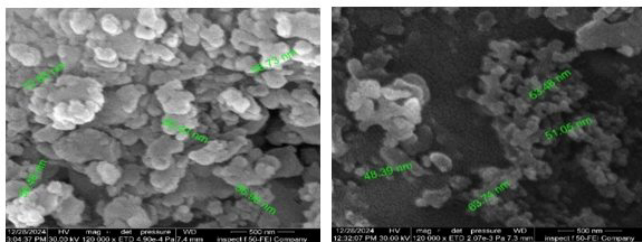


Fig. 3 Field emission scanning electron microscopy (FSEM) of (mSiO₂)

3.4 Factors affecting the adsorption

Effect of mesoporous silica initial concentration on heavy metal adsorption. The impact of initial mesoporous silica concentrations, both commercially available (pure mSiO₂) and laboratory synthesized, was investigated to evaluate adsorption performance toward Pb²⁺, Cd²⁺, and Cr³⁺ ions, as listed in Table 1.

3.5 Effect of initial concentration

To assess the removal of Pb²⁺, Cd²⁺, and Cr³⁺ ions, the effect of initial metal-ion concentration on the adsorption efficiency of mesoporous silica (mSiO₂) was investigated in this study. Using a fixed adsorbent dosage of 0.01 g, experiments were carried out at 298 K and pH 8. Results, summarized in Figure 4, show that the removal efficiency decreases as the initial concentration of metal ions increases. This decrease can be explained by the limited number of adsorption sites on the surface of mSiO₂ relative to the increasing number of metal ions in solution. In contrast, the adsorption capacity (q_e) increased with increasing ion concentration, as more ions were available for uptake on the adsorbent surface. This inverse relationship between removal efficiency and adsorption capacity is consistent with previous findings [28, 29]. Based on the data, 10 mg L⁻¹ was the starting concentration and yielded the highest removal efficiency for all three ions (Pb²⁺, Cd²⁺, and Cr³⁺) at 298 K and pH 8, whereas adsorption capacity was maximized at higher concentrations, such as 100 mg L⁻¹.

The impact of the initial heavy metal-ion concentration on the adsorption rate on the surface of pure mesoporous silica (mSiO₂) prepared from a natural source (porcelanite rock) was also studied to calculate the percentage removal of Pb²⁺, Cd²⁺, and Cr³⁺ ions listed in Table 1.

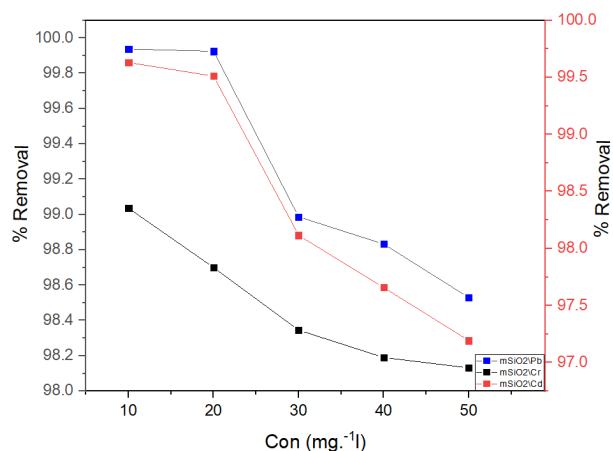


Fig. 4 Effect of the initial concentration on the removal of Pb²⁺, Cd²⁺ and Cr³⁺ ions using mesoporous silica (mSiO₂)

3.6 Impact of pH on heavy metal ion adsorption

Because it directly influences the adsorbent surface charge and adsorption behavior, solution pH is regarded as one of the most important factors affecting the chemical speciation of metal ions in aqueous media, including Pb²⁺, Cd²⁺, and Cr³⁺, as shown in Figure 5. The impact of pH on the adsorption efficiency of mesoporous silica (mSiO₂) was examined in this work, and the optimal pH levels for efficient removal of these ions were determined. Adsorption tests were carried out with 0.01 g of mSiO₂, an initial metal-ion concentration of 100 mg L⁻¹, and a contact time of 2 h at a fixed temperature of 298 K under these fixed conditions. The amount of adsorbent added typically ranges from 0.1 g to 2.0 g per 100-1000 mL of solution, depending on the initial contaminant concentration and the desired adsorbent-to-solution ratio. For example: 0.1 g of mesoporous silica (mSiO₂) in 100 mL solution = 1.0 g L⁻¹ dosage; 1.0 g of mesoporous silica (mSiO₂) in 250 mL solution = 4.0 g L⁻¹ dosage.

The pH of each solution was carefully adjusted to specific values (2, 3, 4, 5, 6, 7, and 8) to examine its influence on removal performance. The percentages removed for Cd²⁺, Pb²⁺, and Cr³⁺ were then calculated for each pH condition to identify the most favorable conditions for adsorption on the silica surface [30]. The

Table 1 Initial concentration and removal values of Pb²⁺, Cd²⁺, and Cr³⁺ ions using pure mesoporous silica mSiO₂ for different concentrations, acid function, contact time, and mass adsorbents

Initial con,mg. (mg.l ¹)	Pb ²⁺ mSiO ₂		Cd ²⁺ mSiO ₂		Cr ³⁺ mSiO ₂		
	Ce (mg.l ⁻¹)	% Removal	Ce (mg.l ⁻¹)	% Removal	Ce (mg.l ⁻¹)	% Removal	
298 K	10	0.006	99.937	0.037	99.630	0.096	99.0360
	20	0.015	99.924	0.098	99.510	0.260	98.7000
	30	0.304	98.986	0.565	98.115	0.497	98.3449
	40	0.467	98.833	0.937	97.657	0.724	98.1908
	50	0.735	98.529	1.403	97.193	0.934	98.1312
298 K	pH	Pb ²⁺ mSiO ₂		Cd ²⁺ mSiO ₂		Cr ³⁺ mSiO ₂	
		Ce (mg.l ⁻¹)	% Removal	Ce (mg.l ⁻¹)	% Removal	Ce (mg.l ⁻¹)	% Removal
	2	6.758	83.329	3.864	87.121	5.001	83.3293
	3	1.489	87.418	2.088	93.040	3.775	87.4183
	4	0.674	91.343	1.359	95.471	2.597	91.3433
	5	0.595	95.678	1.199	96.002	1.297	95.6783
	6	0.402	97.193	1.077	96.410	0.842	97.1933
	7	0.118	99.365	0.520	98.268	0.191	99.3650
8	1.64	99.507	98.496		0.148	99.5077	
298 K	Time (min)	Pb ²⁺ mSiO ₂		Cd ²⁺ mSiO ₂		Cr ³⁺ mSiO ₂	
		Ce (mg.l ⁻¹)	% Removal	Ce (mg.l ⁻¹)	% Removal	Ce (mg.l ⁻¹)	% Removal
	10	0.816	97.12	1.479	95.0707	0.862	97.1260
	20	0.465	97.53	1.376	95.4149	0.464	98.4541
	30	0.327	98.01	1.275	95.7513	0.434	98.5547
	45	0.157	98.9	1.009	96.6377	0.367	98.7783
	60	0.089	97.01	0.987		0.201	99.3310
	90	0.067	96.79	0.770	97.4319	0.123	99.5907
120	0.052	96.2	0.732	97.5600	0.106	99.6459	
298 K	Doses (mg)	Pb ²⁺ mSiO ₂		Cd ²⁺ mSiO ₂		Cr ³⁺ mSiO ₂	
		Ce (mg.l ⁻¹)	% Removal	Ce (mg.l ⁻¹)	% Removal	Ce (mg.l ⁻¹)	% Removal
	0.01	0.054	99.821	1.164	96.1203	0.332	98.8933
	0.02	0.002	99.992	0.558	98.1417	0.157	99.4780
	0.03	0.013	99.957	0.728	97.5727	0.350	98.8350
	0.04	0.099	99.671	0.901	96.9953	0.400	98.6683
0.05	0.144	99.521	0.991	96.6977	0.446	98.5133	

adsorption efficiency of mesoporous silica (mSiO₂) is significantly influenced by solution pH, particularly due to changes in surface charge behavior and electrostatic interactions. At low pH, adsorption sites on the mSiO₂ surface tend to protonate, resulting in a positively charged surface. Under these conditions, the positively charged metal ions (Pb²⁺, Cd²⁺, and Cr³⁺) and the adsorbent surface repel each other electrostatically, thereby hindering access to available adsorption sites. In contrast, within the pH range of 6 to 8, the number of protons in solution decreases, reducing the extent of electrostatic repulsion. As a result, adsorption sites become more accessible, thereby enhancing the interaction between the metal cations and the negatively charged silica surface. Additionally, the increase in hydroxide ions (OH⁻) in this pH range does not compete with metal ions for adsorption sites, further improving metal uptake. These findings align with previous studies [30], confirming that pH 8 provides the most favorable condition for the highest removal efficiency of Pb²⁺, Cd²⁺, and Cr³⁺ ions using mesoporous silica as an adsorbent: Pb²⁺ is 97.8%, Cd²⁺ is 98%, and Cr³⁺ is 99.6%.

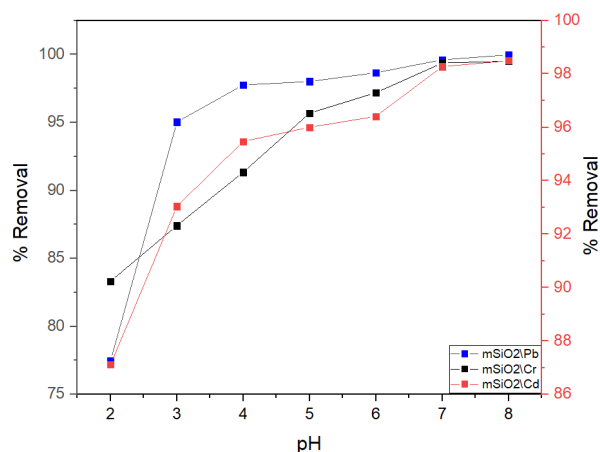


Fig. 5 The impact of pH on the elimination of Pb²⁺, Cd²⁺, Cr³⁺ ions using mesoporous silica (mSiO₂)

3.7 Impact of contact duration on heavy metal ion adsorption

Contact time is a critical factor influencing heavy metal-ion adsorption behavior, and identifying the ideal time frame is crucial for maximizing adsorption efficiency [31]. Assessing the effect of contact duration improves understanding of adsorption dynamics and

helps determine the equilibrium time required to remove metal ions effectively. In general, increasing contact time increases adsorption efficiency for all adsorbents until equilibrium is reached, beyond which no significant change in removal is observed [32].

In this investigation, the removal percentages of Pb^{2+} , Cd^{2+} , and Cr^{3+} ions were calculated under controlled conditions: 298 K, an initial metal-ion concentration of 100 mg L^{-1} , an adsorbent dosage of 0.02 g, and a solution pH of 8. Adsorption was monitored over various contact times of 10, 20, 30, 45, 60, 90, and 120 min, as shown in Figure 6. The collected data were used to assess adsorption rates and determine the equilibrium points for each ion under these fixed conditions.

Adsorption of Pb^{2+} , Cd^{2+} , and Cr^{3+} ions onto mesoporous silica ($mSiO_2$) showed rapid uptake during the first 10 min, followed by a gradual slowdown until equilibrium is reached. Beyond this point, the adsorption rate becomes nearly independent of contact time, indicating saturation of accessible active sites on the adsorbent surface [33]. This behavior can be explained by the large specific surface area of $mSiO_2$ at the start of the process and the abundance of unoccupied adsorption sites. During the initial rapid phase, metal ions interact quickly with the external surface of the silica particles, which is mainly controlled by surface diffusion. As the adsorption rate slows, intra-pore diffusion becomes the dominant mechanism, where ions migrate into the inner porous structure of the material [34, 35]. Based on the experimental results, under the same circumstances, a contact time of 60 min was determined to be optimal, producing the highest effective removal rate for Pb^{2+} , Cd^{2+} , and Cr^{3+} ions.

3.8 Adsorbent mass effect

The impact of different mesoporous silica ($mSiO_2$) dosages on the adsorption of Pb^{2+} , Cd^{2+} , and Cr^{3+} ions was investigated to determine the optimal adsorbent mass for maximum removal efficiency. The tested dosages were 0.01, 0.02, 0.03, 0.04, and 0.05 g. Adsorption tests were carried out at 298 K under constant conditions, with a contact time of 2 h and a solution pH of 8. The results, presented in the table below, illustrate how adsorption capacity and removal percentage vary with increasing adsorbent dosage, providing insight into the optimal $mSiO_2$ dosage for efficient elimination of hazardous metal ions from aqueous solutions.

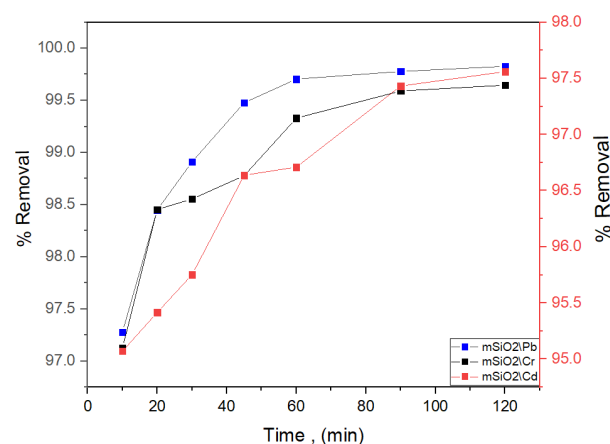


Fig. 6 Impact of contact time the elimination of (Pb^{2+} , Cd^{2+} , and Cr^{3+}) ions using mesoporous silica ($mSiO_2$)

Figure 7 illustrates that, at all tested temperatures, adsorption of Pb^{2+} , Cd^{2+} , and Cr^{3+} ions occurs with high efficiency. The results indicate that increasing the mass of the mesoporous silica adsorbent ($mSiO_2$) enhances the removal of metal ions, primarily due to the corresponding increase in surface area, which provides more active adsorption sites [36, 37]. However, while the adsorbent dosage was varied from 0.01 g to 0.05 g, no significant improvement in removal efficiency was observed beyond a certain point, as shown in Figure 7. This plateau effect is likely due to particle aggregation at higher adsorbent masses, which can reduce the effective surface area and block available active sites, thereby limiting further adsorption capacity [38].

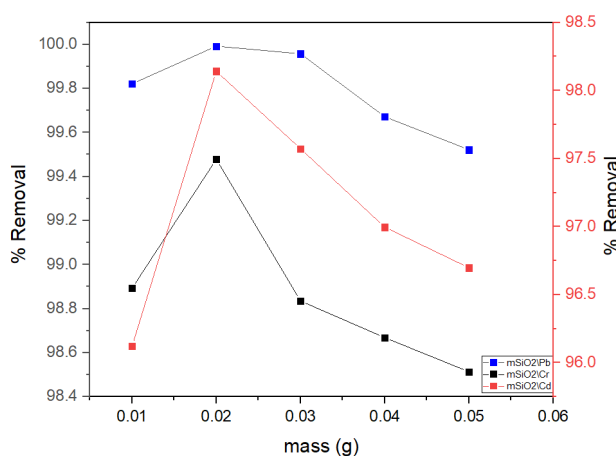


Fig. 7 Adsorbent mass's impact on removal of (Pb^{2+} , Cd^{2+} , and Cr^{3+}) ions using mesoporous silica ($mSiO_2$)

4 CONCLUSION

Natural porcelainite rocks from the western Anbar Governorate of Iraq were successfully used to extract and characterize mesoporous silica (mSiO₂). The material's adsorption-friendly surface and structural characteristics were validated using FTIR, XRD, and FESEM, making it an affordable and effective material for removing harmful heavy metals from aqueous solutions.

The ability of the synthesized mSiO₂ to remove heavy metal ions (Pb²⁺, Cd²⁺, and Cr³⁺) from tainted aqueous solutions was assessed. Adsorption tests were conducted at different initial concentrations (10, 20, 30, 40, and 50 mg L⁻¹), with the temperature maintained at 298 K, pH adjusted to 7, and a contact time of 2 h. The adsorption efficiencies of mSiO₂ were high. Pb²⁺ is 97.8%, Cd²⁺ is 98%, and Cr³⁺ is 99.6%. The material's potential as a selective adsorbent was highlighted by the fact that Cr³⁺ showed the highest removal efficiency among the tested metals. The adsorption efficiencies of mSiO₂ remained high under these conditions, with Pb²⁺ at 97.8%, Cd²⁺ at 98%, and Cr³⁺ at 99.6%.

The ability of the synthesized mSiO₂ to remove heavy metal ions (Pb²⁺, Cd²⁺, and Cr³⁺) from tainted aqueous solutions was also assessed. Adsorption tests were conducted at different starting concentrations (10, 20, 30, 40, and 50 mg L⁻¹), with the temperature kept at 298 K, the pH adjusted to 8, and the contact time fixed at 2 h. The adsorption efficiencies of mSiO₂ were high. Pb²⁺ is 97.8%, Cd²⁺ is 98%, and Cr³⁺ is 99.6%. The material's potential as a selective adsorbent was highlighted by Cr³⁺, showing the highest removal efficiency among the tested metals.

The ability of the synthesized mSiO₂ to remove heavy metal ions (Pb²⁺, Cd²⁺, and Cr³⁺) from tainted aqueous solutions was assessed under the same starting concentrations (10, 20, 30, 40, and 50 mg L⁻¹), with temperature kept at 298 K, pH adjusted to 8, and contact time fixed at 2 h. The adsorption efficiencies of mSiO₂ were high: Pb²⁺ is 97.8%, Cd²⁺ is 98%, and Cr³⁺ is 99.6%. Based on the high correlation coefficient (R²), the results suggest monolayer adsorption behavior on a comparatively homogeneous surface.

Acknowledgement

N/A

Funding source

No funds received.

Data availability

N/A

DECLARATIONS

Conflict of interest

The authors declare no competing interests.

Consent to publish

N/A

Ethical approval

N/A

REFERENCES

- [1] Barani S, Sebastian SP, Dhevagi P, Prasanthrajan M, Suganthi A. Synthesis of silica nanoparticles (SiNPs) from agro-wastes for removal of heavy metals from an aqueous medium – a mini review. *Green Chemistry Letters and Reviews*. 2024;17(1):2422416. [10.1080/17518253.2024.2422416](https://doi.org/10.1080/17518253.2024.2422416)
- [2] Liu ZS, Li WK, Huang CY. Synthesis of mesoporous silica materials from municipal solid waste incinerator bottom ash. *Waste Management*. 2014;34(5):893-900. [10.1016/j.wasman.2014.02.016](https://doi.org/10.1016/j.wasman.2014.02.016)
- [3] Popova M, Mitova V, Dimitrov M, Rosmini C, Tsacheva I, Shestakova P, et al. Mesoporous silica with an alveolar construction obtained by eco-friendly treatment of rice husks. *Molecules*. 2024;29(15):3540. [10.3390/molecules29153540](https://doi.org/10.3390/molecules29153540)
- [4] Dassekpo JBM, Zhong H, Iong C, Dong Z, Shao N, Dassekpo IS, et al. Waste glass as a source for green synthesis of mesoporous adsorbent for efficient removal of heavy metals. *Microporous and Mesoporous Materials*. 2025;383:113393. [10.1016/j.micromeso.2024.113393](https://doi.org/10.1016/j.micromeso.2024.113393)
- [5] Ezzeddine Z, Batonneau-Gener I, Ghssein G, Pouilloux Y. Recent advances in heavy metal adsorption via organically modified mesoporous silica: A review. *Water*. 2025;17(5):669. [10.3390/w17050669](https://doi.org/10.3390/w17050669)
- [6] Li G, Zhao Z, Liu J, Jiang G. Effective heavy metal removal from aqueous systems by thiol functionalized magnetic mesoporous silica. *Journal of Hazardous Materials*. 2011;192(1):277-83. [10.1016/j.jhazmat.2011.05.015](https://doi.org/10.1016/j.jhazmat.2011.05.015)
- [7] Samani M, Ahlawat YK, Golchin A, Alikhani HA, Fathi-Gerdeldani A, Ahlawat U, et al. Nano

- silica-mediated stabilization of heavy metals in contaminated soils. *Scientific Reports*. 2024;14:20496. [10.1038/s41598-024-69182-0](https://doi.org/10.1038/s41598-024-69182-0)
- [8] Abdulzahra MA, Adam RW, Egzar HK. New poly-tetrazole synthesis and adsorption surface investigation based on sulfadiazine grafted polyimine. *Chemistry, Physics and Technology of Surface*. 2024;15(3):420-8. [10.15407/hftp15.03.420](https://doi.org/10.15407/hftp15.03.420)
- [9] Dinker MK, Kulkarni PS. Recent advances in silica-based materials for the removal of hexavalent chromium: A review. *Journal of Chemical & Engineering Data*. 2015;60(9):2521-40. [10.1021/acs.jced.5b00292](https://doi.org/10.1021/acs.jced.5b00292)
- [10] Grozdov D, Zinicovscaia I. Mesoporous materials for metal-laden wastewater treatment. *Materials*. 2023;16(17):5864. [10.3390/ma16175864](https://doi.org/10.3390/ma16175864)
- [11] Wadhawan S, Jain A, Nayyar J, Mehta SK. Role of nanomaterials as adsorbents in heavy metal ion removal from waste water: A review. *Journal of Water Process Engineering*. 2020;33:101038. [10.1016/j.jwpe.2019.101038](https://doi.org/10.1016/j.jwpe.2019.101038)
- [12] Gong Z, Chan HT, Chen Q, Chen H. Application of nanotechnology in analysis and removal of heavy metals in food and water resources. *Nanomaterials*. 2021;11(7):1792. [10.3390/nano11071792](https://doi.org/10.3390/nano11071792)
- [13] Flores D, Almeida CMR, Gomes CR, Balula SS, Granadeiro CM. Tailoring of mesoporous silica-based materials for enhanced water pollutants removal. *Molecules*. 2023;28(10):4038. [10.3390/molecules28104038](https://doi.org/10.3390/molecules28104038)
- [14] Abuhatab S, El-Qanni A, Al-Qalaq H, Hmoudah M, Al-Zerei W. Effective adsorptive removal of Zn²⁺, Cu²⁺, and Cr³⁺ heavy metals from aqueous solutions using silica-based embedded with NiO and MgO nanoparticles. *Journal of Environmental Management*. 2020;268:110713. [10.1016/j.jenvman.2020.110713](https://doi.org/10.1016/j.jenvman.2020.110713)
- [15] Zhang P, Yang M, Lan J, Huang Y, Zhang J, Huang S, et al. Water quality degradation due to heavy metal contamination: Health impacts and eco-friendly approaches for heavy metal remediation. *Toxics*. 2023;11(10):828. [10.3390/toxics11100828](https://doi.org/10.3390/toxics11100828)
- [16] Holmes SM, Zholobenko VL, Thursfield A, Plaisted RJ, Cundy CS, Dwyer J. In situ FTIR study of the formation of MCM-41. *Journal of the Chemical Society, Faraday Transactions*. 1998;94(14):2025-32. [10.1039/A801898G](https://doi.org/10.1039/A801898G)
- [17] Liu D, Qin S, Qiao H, Xing X, Yan K. Comparative study on adsorption performance of Pb(II) using fly ash-based mesoporous silica through sol-gel method with different aging temperature. *Microporous and Mesoporous Materials*. 2025;387:113523. [10.1016/j.micromeso.2025.113523](https://doi.org/10.1016/j.micromeso.2025.113523)
- [18] Li S, Li S, Wen N, Wei D, Zhang Y. Highly effective removal of lead and cadmium ions from wastewater by bifunctional magnetic mesoporous silica. *Separation and Purification Technology*. 2021;265:118341. [10.1016/j.seppur.2021.118341](https://doi.org/10.1016/j.seppur.2021.118341)
- [19] Zhao XS, Lu GQ. Modification of MCM-41 by Surface Silylation with Trimethylchlorosilane and Adsorption Study. *The Journal of Physical Chemistry B*. 1998;102(9):1556-61. [10.1021/jp972788m](https://doi.org/10.1021/jp972788m)
- [20] Wieszczycka K, Filipowiak K, Wojciechowska I, Buchwald T, Siwińska-Ciesielczyk K, Strzemińska B, et al. Novel highly efficient ionic liquid-functionalized silica for toxic metals removal. *Separation and Purification Technology*. 2021;265:118483. [10.1016/j.seppur.2021.118483](https://doi.org/10.1016/j.seppur.2021.118483)
- [21] Rahman ML, Sarjadi MS, Arshad SE, Mustafa B, Heffeman MA, O'Reilly EJ, et al. Synthesis of silica-supported hydroxamic ligand for removal of metals ions from water. *Journal of Nanoscience and Nanotechnology*. 2021;21(3):1570-7. [10.1166/jnn.2021.18982](https://doi.org/10.1166/jnn.2021.18982)
- [22] Aziz KHH, Mustafa FS, Omer KM, Hama S, Hamarawf RF, Rahman KO. Heavy metal pollution in the aquatic environment: Efficient and low-cost removal approaches to eliminate their toxicity: A review. *RSC Advances*. 2023;13:17595-610. [10.1039/D3RA00723E](https://doi.org/10.1039/D3RA00723E)
- [23] Paukshtis EA, Yaranova MA, Batueva IS, Bal'zhinimaev BS. A FTIR study of silanol nests over mesoporous silicate materials. *Microporous and Mesoporous Materials*. 2019;288:109582. [10.1016/j.micromeso.2019.109582](https://doi.org/10.1016/j.micromeso.2019.109582)
- [24] Briffa J, Sinagra E, Blundell R. Heavy metal pollution in the environment and their toxicological effects on humans. *Heliyon*. 2020;6(9):e04691. [10.1016/j.heliyon.2020.e04691](https://doi.org/10.1016/j.heliyon.2020.e04691)
- [25] Singh V, Singh N, Rai SN, Kumar A, Singh AK, Singh MP, et al. Heavy metal contamination in the aquatic ecosystem: Toxicity and its remediation using eco-friendly approaches. *Toxics*. 2023;11(2):147. [10.3390/toxics11020147](https://doi.org/10.3390/toxics11020147)

- [26] Tchounwou PB, Yedjou CG, Patlolla AK, Sutton DJ. Heavy metal toxicity and the environment. In: Molecular, Clinical and Environmental Toxicology. vol. 101 of *Experientia Supplementum*. Springer; 2012. p. 133-64. [10.1007/978-3-7643-8340-4_6](https://doi.org/10.1007/978-3-7643-8340-4_6)
- [27] Tella JO, Ajanaku KO, Adekoya JA, Banerjee R, Patra CR, Pavuluri S, et al. Physicochemical and textural properties of amino-functionalised mesoporous silica nanomaterials from different silica sources. *Results in Chemistry*. 2024;7:101505. [10.1016/j.rechem.2024.101505](https://doi.org/10.1016/j.rechem.2024.101505)
- [28] Saod WM, Alaallah NJ, Abdulkareem EA, Hilal NN, AlBiajawi MI. Study of effective removal of nickel and cobalt from aqueous solutions by FeO@mSiO₂ nanocomposite. *Results in Chemistry*. 2025;13:101992. [10.1016/j.rechem.2024.101992](https://doi.org/10.1016/j.rechem.2024.101992)
- [29] Da'na E. Adsorption of heavy metals on functionalized-mesoporous silica: A review. *Microporous and Mesoporous Materials*. 2017;247:145-57. [10.1016/j.micromeso.2017.03.050](https://doi.org/10.1016/j.micromeso.2017.03.050)
- [30] Saod WM, Oliver IW, Thompson DF, Contini A, Zholobenko V. Zinc oxide – mesoporous silica nanocomposite: preparation, characterisation and application in water treatment for lead, cadmium and chromium removal. *International Journal of Environmental Analytical Chemistry*. 2024;104(20):9772-84. [10.1080/03067319.2023.2246016](https://doi.org/10.1080/03067319.2023.2246016)
- [31] Petreanu I, Niculescu VC, Enache S, Iacob C, Teodorescu M. Structural characterization of silica and amino-silica nanoparticles by Fourier transform infrared (FTIR) and Raman spectroscopy. *Analytical Letters*. 2023;56(2):390-403. [10.1080/00032719.2022.2083144](https://doi.org/10.1080/00032719.2022.2083144)
- [32] Saod WM, Oliver IW, Contini A, Zholobenko V. Synthesis and characterisation of an iron oxide mesoporous silica nano-composite and its application in removal of methylene blue dye. *Journal of Molecular Structure*. 2025;1319:139390. [10.1016/j.molstruc.2024.139390](https://doi.org/10.1016/j.molstruc.2024.139390)
- [33] Sarker A, Al Masud MA, Deepo DM, Das K, Nandi R, Ansary MWR, et al. Biological and green remediation of heavy metal contaminated water and soils: A state-of-the-art review. *Chemosphere*. 2023;332:138861. [10.1016/j.chemosphere.2023.138861](https://doi.org/10.1016/j.chemosphere.2023.138861)
- [34] Zhao J, Zhu YJ, Wu J, Zheng JQ, Zhao XY, Lu BQ, et al. Chitosan-coated mesoporous microspheres of calcium silicate hydrate: Environmentally friendly synthesis and application as a highly efficient adsorbent for heavy metal ions. *Journal of Colloid and Interface Science*. 2014;418:208-15. [10.1016/j.jcis.2013.12.016](https://doi.org/10.1016/j.jcis.2013.12.016)
- [35] Dinari M, Soltani R, Mohammadnezhad G. Kinetics and thermodynamic study on novel modified-mesoporous silica MCM-41/polymer matrix nanocomposites: Effective adsorbents for trace Cr(VI) removal. *Journal of Chemical & Engineering Data*. 2017;62(8):2316-29. [10.1021/acs.jced.7b00197](https://doi.org/10.1021/acs.jced.7b00197)
- [36] Mortada WI, Nabieh KA, Helmy TE, Abou El-Reash YG. Microwave-assisted synthesis of MCM-41 composite with rice husk and its functionalization by dithizone for preconcentration of some metal ions from water and food samples. *Journal of Food Composition and Analysis*. 2022;106:104352. [10.1016/j.jfca.2021.104352](https://doi.org/10.1016/j.jfca.2021.104352)
- [37] Song Y, Huang P, Li H, Li R, Zhan W, Du Y, et al. Uptake of arsenic(V) using iron and magnesium functionalized highly ordered mesoporous MCM-41 (Fe/Mg-MCM-41) as an effective adsorbent. *Science of the Total Environment*. 2022;833:154858. [10.1016/j.scitotenv.2022.154858](https://doi.org/10.1016/j.scitotenv.2022.154858)
- [38] Taleb MA, Kumar R, Barakat MA, Almeelbi T, Seliem MK, Ahmad A. Recent advances in heavy metals uptake by tailored silica-based adsorbents. *Science of The Total Environment*. 2024;955:177093. [10.1016/j.scitotenv.2024.177093](https://doi.org/10.1016/j.scitotenv.2024.177093)

How to cite this article

Hussein SH, Saod WM. Rocks as a sustainable resource mSiO₂: Extraction, characterisation, and use for heavy metals removal from wastewater by adsorption process. *Journal of University of Anbar for Pure Science*. 2026; 20(1):155-164. doi:[10.37652/juaps.2025.160965.1403](https://doi.org/10.37652/juaps.2025.160965.1403)