



Drinking Water Treatment Plant's Sludge as an Adsorbent for Removal of Heavy Metals: Effects of Contact Time, Adsorbent Dosage, and pH Level

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Abstract: Heavy metals such as chromium, lead, copper, iron, manganese, and vanadium are several common heavy metals that can enter the environment through industrial activities, wastewater plants, and much more. A high concentration of these metals may cause severe health issues for living things. To address this issue, the present study reports the removal of chromium (Cr) and lead (Pb) metal ions from aqueous solution by activated sludge as an adsorbent. The effects of contact time (15–120 min), adsorbent dosage (40–160 g/L), and pH level (2–11) were studied in a batch experiment. The adsorbents were characterized using an X-ray fluorescence spectrometer (XRF), a Fourier Transform Infrared (FTIR) spectrometer, and a Scanning Electron Microscope (SEM). Adsorption results showed that the adsorption capacity of Cr(III) and Pb(II) ions increased with increasing contact time, adsorbent dosage, and pH. The variation in adsorbent dosage showed the highest adsorption capacities for Cr(III) and Pb(II) ions, 4.13 mg/g and 4.91 mg/g, respectively, at pH 5, an adsorbent dosage of 40 g/L, and a contact time of 60 min. These findings show that sludge from drinking water treatment plants can be effectively and sustainably used as an adsorbent to remove heavy metals. These findings also encourage the reuse of local waste materials and help in creating new environmental technologies.

Keywords: Adsorbent; adsorption; chromium; lead; drinking water treatment plant

1. Introduction

Access to clean water is considered one of the most important humanitarian priorities. In many industries, clean water plays an important role and is essential to feedstock [1]. However, the rapid activities of industry itself also contribute to the release of pollutants to the environment, such as heavy metals. Heavy metals such as chromium, lead, copper, iron, manganese, and vanadium are among the most common that can enter the environment through industrial activities, wastewater treatment plants, mining processes, and other sources [2]. It has become concerning because high concentrations of these metals can cause severe health problems for living organisms. Therefore, researchers have been working and developing various methods of remediation to prevent its further contamination [3–4]. There are three main methods of heavy metal remediation: physical, chemical, and biological methods. Various methods, such as chemical precipitation, adsorption, reverse osmosis, and

biological treatments, are technologies that have been developed to reduce heavy metals in wastewater [5–6]. Each method has its own advantages and disadvantages when handling specific metal ions. Among them, adsorption has been widely applied due to its benefits such as high effectiveness, simple equipment requirements, cost-effectiveness, and the capability to remove a variety of pollutants [7–8].

Generally, adsorbents can be grouped into four categories: natural-source adsorbents, polymeric adsorbents, adsorbents derived from industrial by-products, and carbon-nanomaterial-based adsorbents [9]. Adsorption is a method used to separate substances by removing pollutants from one phase, such as a liquid or gas, and concentrating them on the surface of an adsorbent. This process is commonly used across various industries, including dyes, paper, cosmetics, textiles, and others, to eliminate harmful organic compounds such as pharmaceuticals, endocrine disruptors, and organic and inorganic contaminants from water. Several adsorbents have proven effective for removing heavy metals from solution [10].

The water treatment process generates millions of tons of solid waste, and the amount of sludge increases as water use rises. Dry residue from water treatment is produced every day; however, managing sludge from water treatment plants remains challenging for all water authorities. Recovering, reusing, and recycling sludge are now necessary for water utilities seeking to manage resources sustainably [11]. Nowadays, there is a growing emphasis on material circularity, which involves converting waste into valuable added materials through more sustainable methods. Significant efforts have been made to develop adsorbents from renewable and low-cost biomass sources. Many studies have shown that water treatment sludge can effectively act as a coagulant and adsorbent for pollutants. Bensitel et al. [12] have focused on the experiment that used sludge to treat raw water, and it was found that adding sludge reduced the levels of chloride (Cl^-) and sulfate (SiO_4^{2-}) ions, and this finding showed that sludge can act effectively as a coagulant in the treatment process [12]. Singh & Garg [13] synthesized hydrochar from hydrothermally pretreated sewage sludge and centrifuged sewage sludge as an adsorbent for methylene blue from synthetic wastewater. They found that dye removal reached 98.3% with KOH-activated hydrochar [13]. Other researchers have investigated biochar prepared from sludge by pyrolysis for the removal of lead (Pb^{2+}), methyl orange (MO), and ciprofloxacin (CIP) from wastewater. The investigation results showed that the biochar from sludge exhibited a high adsorption capacity. Furthermore, the Langmuir isotherm and Pseudo-second-order model indicated that the adsorption capacity of MgAl-layered double hydroxides (Mg-Al LDO) for MO was 18.6% higher than for CIP. Mg-Al LDO can rapidly remove Pb^{2+} from wastewater with an adsorption capacity of 147.89 mg/g [14]. Alum sludge from drinking water production was used as an adsorbent for wastewater treatment in the textiles industry, and the results showed that the adsorption capacity is strongly dependent on initial dye concentration, initial pH, and the adsorbent. On the other hand, the maximal removal of an anion dye was 97% at pH 2 [11].

Even though many studies have shown that sludge is adsorbent, the application of activated water treatment sludge for water purification or contaminant removal from wastewater is not yet widely adopted. Therefore, this study aimed to assess the feasibility of using adsorbent material from a drinking water treatment plant's sludge for the adsorption of chromium (Cr) and lead (Pb) from aqueous solution, and to investigate the effects of contact time, adsorbent dosage, and pH on adsorption capacity.

2. Materials and Methods

2.1 Preparation of adsorbent

The sludge was collected from the drinking water treatment plant in Palembang, Indonesia. The sludge was washed using distilled water to remove the impurities, then dried in an oven at 120 °C for 8 h. The dried sludge was ground in a mortar into a fine powder and separated into particle sizes using a 200-mesh sieve. The prepared sludge was stored in plastic containers to prevent moisture reabsorption. Before use in adsorption experiments, the sludge was activated with 2 M sodium hydroxide (NaOH). The activation was prepared as follows: the required amount of dried sludge was mixed with 2 M NaOH (ratio 1:2) and stirred at room temperature for 3 h. The mixture was then filtered, and the solid was washed with distilled water until the pH was 7 (8). The solid was then dried in an oven at 120 °C for 8 h and ground using a mortar. The activated sludge was ready to use as an adsorbent.

2.2 Chromium and lead adsorption experiment

Standard chromium (Cr) and lead (Pb) solutions were obtained by dissolving chromium nitrate ($\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, Merck) and lead (II) nitrate ($\text{Pb}(\text{NO}_3)_2 \cdot 9\text{H}_2\text{O}$, Merck) salts in calculated stoichiometric amounts in distilled water to prepare solutions of 300 mg/L of each metal. To study the effect of pH on the adsorption, 1 M HCl and 1 M NaOH solutions were added to the synthetic solution.

2.3 Preparation of aqueous solution

The effects of contact time (15, 30, 60, 90, and 120 min), adsorbent dosage (40, 80, 120, and 160 g/L), and pH (2, 4, 5, 7, 9, and 11) were investigated in a batch experiment. To study the effect of pH, 2 g of adsorbent was added to 50 mL of a single-metal standard solution (Cr or Pb), and the pH was adjusted to 2 with 1 M HCl. The mixture was stirred at 120 rpm for 30 min at room temperature, then filtered using filter paper. The metal ion concentration in the filtrate was measured by an atomic absorption spectrophotometer (AAS) (ICE 3000 Series Atomic Absorption Thermo Fisher Scientific Spectrometers). The same procedure was applied to other variations in pH and contact time. 1 M NaOH was used to adjust the pH to basic. The equilibrium adsorption capacity (Q_e , mg/g) of the metal ion was calculated using Eqs. (1) [11]:

$$Q_e = \frac{C_0 - C}{m} \times V \quad (1)$$

where C_0 and C are the initial and final concentrations (mg/L) of the metal solutions; m is the adsorbent dosage (g), and V is the volume of solution (L).

2.4 Adsorbent characterization

The adsorbent was characterized using various analytical techniques. The adsorbent was chemically analyzed using an energy X-Ray Fluorescence Spectrometer (XRF) (HD Prime Analyzer, XOS) to determine the elemental composition. Scanning Electron Microscope (SEM) (TSCAN VEGA3) for morphological analysis of the adsorbent and Fourier Transform Infra-Red (FTIR) (Shimadzu) for functional group determination.

3. Results and Discussion

3.1 Effect of contact time on metal ion adsorption

All experiments were conducted in triplicate, and data were expressed as mean $\pm$ standard deviation. Contact time may affect the adsorption process, and it is an important parameter. The equilibrium adsorption capacity of sludge-based adsorbents varies with the time that the adsorbent is in contact with metal ions in aqueous solution. The equilibrium adsorption capacity of Cr(III) and Pb(II) ions using 40 g/L adsorbent and pH 5 in the range of 15–120 min, and the concentration of the remnant metal was measured using AAS. The equilibrium adsorption capacity of Cr(III) and Pb(II) ions as a function of contact time is presented in Figure 1. It was observed that the equilibrium adsorption capacity increases with increasing contact time. The rapid metal adsorption in the initial stage of contact time is attributed to the large number of available active sites, with adsorption capacities of 1.51 and 2.25 mg/g for Cr(III) and Pb(II) ions, respectively. As the contact time was increased from 60 to 90 min, the adsorption capacity continued to increase at a relatively slower rate. This indicates that many active sites were already occupied by metal ions, reducing the number of open adsorption sites. As a result, the adsorption process slowly approached equilibrium. At 105–120 min, the adsorption capacities of Cr(III) and Pb(II) ions became almost constant. The equilibrium capacity at 120 min was 6.47 mg/g for Cr(III) and 7.13 mg/g for Pb(II) ions. The findings further indicate that the ability to absorb Pb(II) ions was consistently greater than that for Cr (III) ions throughout the entire contact time. This variation might be linked to differences in ionic radius, hydration energy, electronegativity, and the metal ions' attraction to the functional groups on the adsorbent surface. Pb(II) ions typically demonstrate stronger interactions with oxygen-containing functional groups, such as hydroxyl and carboxyl groups, which leads to improved adsorption performance compared to Cr(III) ions [15–16].

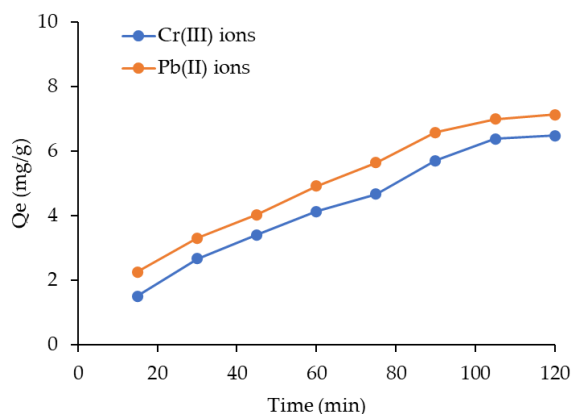


Figure 1. Effect of contact time on adsorption capacity

The rapid adsorption of Pb(II) ions onto the adsorbent has been studied by several researchers. Ghorpade & Ahammed (2018) have evaluated the removal of heavy metals from an electroplating wastewater using an adsorbent derived from water treatment sludge, and applied it for the adsorption of heavy metals such as Cu(II), Co(II), Pb(II), Cr(II), Zn(II), and Hg(II). They examined adsorption on 1 g of adsorbent over 15–480 min and found that the percentage removal of Pb was the highest among the others, since Pb could form strong inner-sphere complexes with aluminum and iron oxide surfaces, which may be responsible for the faster adsorption observed. In mineral and activated carbon-based adsorbents, adsorption is driven by a combination of electrostatic forces, ion exchange, and surface complexation. This is evidenced by the shift in wavenumber, the emergence of new peaks, and changes in intensity in the FTIR spectrum after adsorption as reported by Saenplee et al. (2025) [17–19]. The trend of rapid contact time reaching equilibrium within 60 minutes, and a decrease in performance with increasing time, is also typical of surface adsorption driven by hydroxyl and carboxyl groups, as reported in Si-Al-Fe-based composite materials

3.2 Effect of adsorbent dosage on metal ion adsorption

The adsorbent dosage is an important factor that affects the adsorbate's adsorption percentage. To study the effect of adsorbent dosage, it was varied from 40 g/L to 160 g/L for a 300 mg/L aqueous solution of each Cr and Pb ion. The solutions were stirred at pH 5 for 60 min, and AAS was used to measure the residual metal concentration.

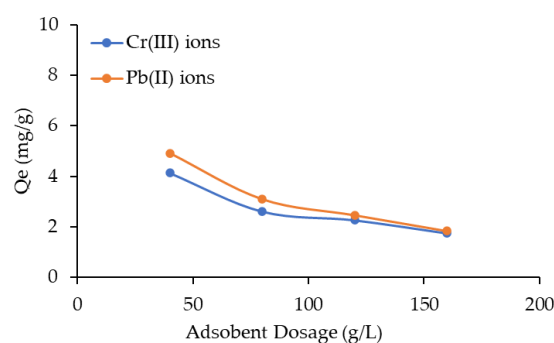


Figure 2. Effect of adsorbent dosage on adsorption capacity

Results presented in Figure 2 revealed that the adsorption capacity decreased gradually with increasing adsorbent dosage from 40 g/L to 160 g/L. At an adsorbent dosage of 40 g/L, the adsorption capacities were 4.13 mg/g for Cr(III) and 4.91 mg/g for Pb(II). However, the adsorption capacities decreased as the adsorbent dosage increased to 80, 120, and 160 g/L, reaching 1.73 (1.82 mg/g) at the highest adsorbent concentration. This trend shows that using more adsorbent does not always increase the adsorption capacity per unit mass of adsorbent. The decrease in adsorption capacity with increasing adsorbent dosage can be

explained by the fact that adsorption sites become saturated. When a lot of adsorbent is added, the number of sites where it can bind to metal ions becomes much larger than the number of metal ions in the solution. As a result, many adsorption sites remain empty, leading to less metal being absorbed per gram of adsorbent. The metal ions present spread across a larger volume of adsorbent material, leading to a reduction in the measured adsorption capacity (q). This could be associated with the formation of aggregation resulting from inter-particle interaction as observed by Wołowiec et al. (2019) and Intachai et al. (2022) [20–21].

3.3 Effect of pH on metal ion adsorption

The pH of the adsorbate solution influences the adsorption capacity of metal ions by altering the adsorbent's surface charge. Therefore, in this study, the pH of the solutions needs to be optimized to maximize the adsorption of Cr(III) and Pb(II) ions. The aqueous solutions of Cr and Pb were prepared at 6 different pH values (2, 4, 5, 7, 9, and 11) using dilute HCl and NaOH solutions, as described in the methodology. The adsorption process was carried out at a 40 g/L adsorbent dosage and a contact time of 60 min for each metal ion solution.

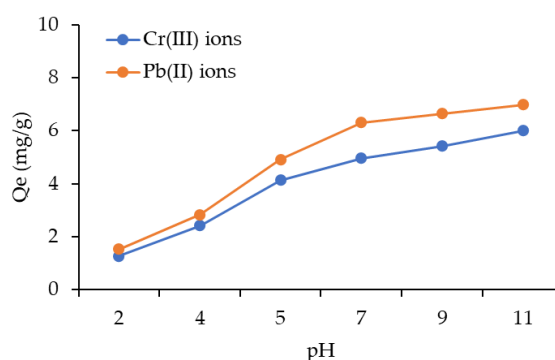


Figure 3. Effect of pH level on adsorption capacity

As shown in Figure 3, the adsorption capacity of Cr(III) and Pb(II) ions increased with an increase in pH of each metal solution. The adsorption capacity of Cr(III) and Pb(II) ions was 1.26 mg/g and 1.52 mg/g, respectively, at pH 2 and reached the maximum adsorption capacity of 6.01 mg/g and 6.99 mg/g, respectively, at pH 11. The pH of the aqueous metal solution plays an important role in the adsorption of metal ions by the adsorbents. The pH study demonstrated that the adsorption efficiency increased as the solution pH increased from acidic to alkaline conditions. Based on the literature, hydrogen ions can compete with metal ions for adsorption sites, and the chemical forms of the metal ions vary with pH. At higher pH values, the adsorbent's functional groups lose protons, rendering the surface negatively charged. This negative charge attracts positively charged Cr^{3+} and Pb^{2+} metal ions, allowing them to attach through electrostatic forces. On the other hand, when the metal solutions are acidic, the functional groups on the adsorbent gain protons, resulting in a positive charge and the formation of metal-ion-adsorbent complexes, as the primary binding sites are not effectively engaged [22]. However, at alkaline pH levels, particularly when they exceed pH 6 to 7, it is important to consider the role of hydroxide precipitation. Cr(III) and Pb(II) ions can form insoluble hydroxide compounds, such as $\text{Cr}(\text{OH})_3$ and $\text{Pb}(\text{OH})_2$, at high pH, thereby increasing adsorption capacity. Therefore, the observed metal adsorption capacity in alkaline conditions might be due to a combination of adsorption and precipitation processes, rather than adsorption alone [23, 24].

The removal of metal ions by electrostatic attraction was also studied by Du et al. (2020). They reported that the removal efficiency for Cd(II), Cu(II), and Zn(II) using a granular sludge-clay (GSC) adsorbent increased with increasing pH. They described the protonation of the adsorbent surface at pH below 4, providing positively charged sites, which are responsible for the low removal efficiency of metal ions [23]. Other studies have shown that at high pH levels, the amount of H^+ decreases and the adsorbent surface becomes negatively charged; therefore, it attracts more positive metal ions available in the solution, and the other possibility is the formation of hydrolyzed complexes that might be generated at high pH values, which

increase the adsorption capacity [24]. Other researchers have reported similar observations: increased removal of cationic metals such as Cu(II), Cd(II), and Zn(II) at higher pH values [25].

3.4 X-Ray Fluorescence Spectrometer (XRF)

XRF determined the elemental content of the adsorbent, and the results are shown in Table 1. It can be seen that sludge from a drinking water treatment plant is mainly composed of silicon (Si), aluminum (Al), and iron (Fe). The high Si content (41.03%) could be associated with the presence of sand and clayey substances in drinking water treatment sludge, and Al (25.10%) is attributed to the use of aluminum sulfate as a coagulant in the water treatment process [26]. The Fe content (6.37%) in the adsorbent may be due to the leaching of iron during the coagulation process. Other elements, such as calcium (Ca), magnesium (Mg), and potassium (K), were detected at low levels.

Table 1. XRF results of the adsorbent.

Component	Content (%)
SiO ₂	41.03
Al ₂ O ₃	25.10
Fe ₂ O ₃	6.37
CaO	1.92
MgO	1.81
K ₂ O	1.13
TiO ₂	3.2
Na ₂ O	2.8
P ₂ O ₅	2.4
SO ₃	1.8
MnO	0.54
Organic matter	11.63

3.5 Scanning Electron Microscope (SEM)

The morphology of the adsorbent was examined using scanning electron microscopy (SEM). The surface morphologies of the adsorbents before and after adsorption are shown in Figure 4. Before use in adsorption (Fig. 4a), the adsorbent surface appeared to have irregular structures, heterogeneous pores, and a rough surface. The adsorbent surface has many shapes, providing active sites for metal ion adsorption. SEM examination of adsorbents after adsorption of Cr and Pb metal ions shows similar appearances. As shown in Figures 4b and 4c, the adsorbent surfaces exhibited agglomerated structures and overlapping regions due to the adsorption of Cr and Pb metal ions; this appearance is even clearer on the adsorbent after Pb adsorption.

3.6 Fourier Transform Infra-Red (FTIR)

The characteristic functional groups of adsorbents were analyzed using FTIR. The spectra were captured in the frequency range from 4000 to 500 cm⁻¹. Figure 5a shows the FTIR spectra of adsorbents before and after adsorption of Cr metal ions, while Figure 5b shows the FTIR spectra of adsorbents before and after adsorption of Pb metal ions. As shown in Figure 5, the FTIR spectra of the adsorbents exhibit a high degree of similarity, as they share the same functional bands. The spectrum obtained shows that the main groups present in the adsorbents are carbonyl, hydroxyl, amine, and double-bond groups. Two small peaks between 4000 cm⁻¹ and 3500 cm⁻¹ are linked to the stretching vibration of the O–H bond in alcohols, phenols, and secondary N–H) amines [27]. FTIR spectrum of the adsorbent before adsorption of Cr metal ions represents a small peak almost linear at 3401.88 cm⁻¹, indicating the stretching vibration of O–H bond in hydroxyl group which was broaden in the adsorbent after adsorption of Cr metal ions, suggesting an increased number of hydroxyl functional groups and implying that the surface complexation reactions took place between Cr metal ions and aluminum oxide in the adsorbent.

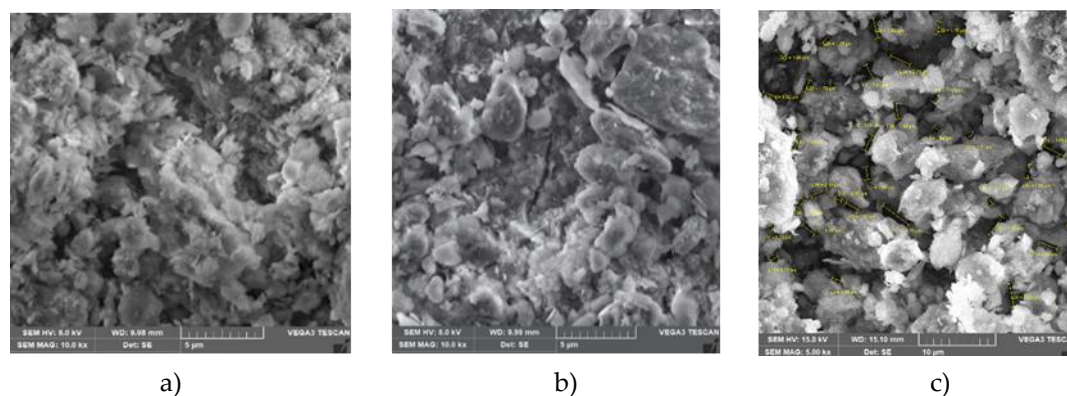


Figure 4. (a) SEM image of adsorbent before adsorption test at 500× magnification, scale bar = 100 μm, (b) SEM image of adsorbent after Cr(III) ions adsorption at 2000× magnification, scale bar = 20 μm, (c) SEM image of adsorbent after Pb(II) ions adsorption at 5000× magnification, scale bar = 10 μm

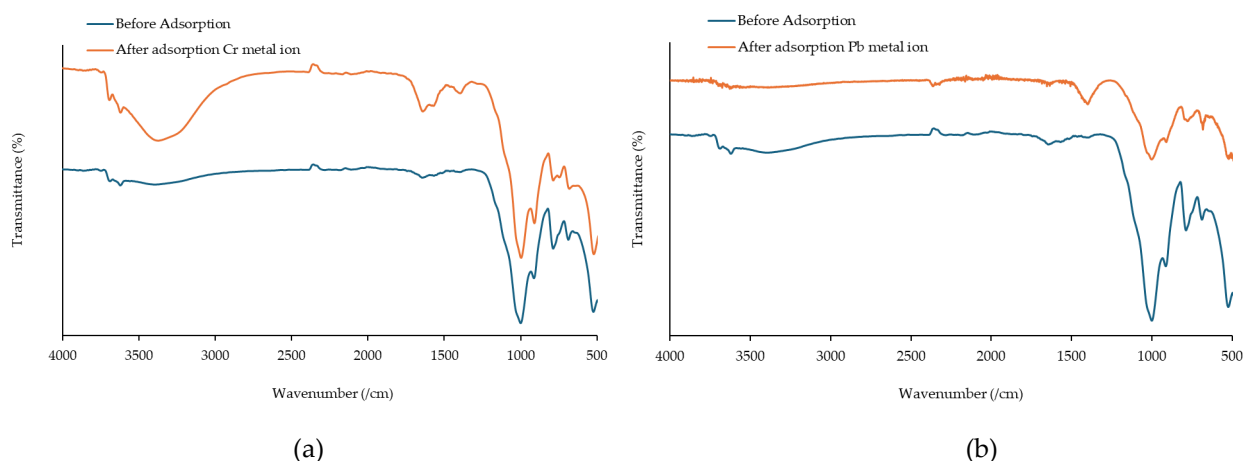


Figure 5. FTIR spectra of adsorbents: (a) Cr metal ions, (b) Pb metal ions

The band at 1638.18 cm^{-1} , which is clearly observed in the adsorbent after adsorption of Cr metal ions, is assigned to vibrational bending of H–O–H [26]. The bending vibration of water physically held on the surface and the structural hydroxyl groups within the mineral structure (Si–OH, Al–OH, Fe–OH), which are characteristic features of hydrated aluminosilicate and iron-based materials. This observation aligns with the elemental composition revealed through XRF analysis. Figure 5a and 5b show a sharp, small band at 1392.88 cm^{-1} , which shifted to 1397.91 cm^{-1} in the adsorbent after adsorption of Cr and Pb metal ions, respectively. This band is assigned to –OH deformation vibrations of metallic oxides), suggesting that the adsorbent adsorbed Cr and Pb metal ions. The shift in wavenumber corresponds to a change in the energy of the functional group, suggesting the involvement of carboxyl and hydroxyl groups in the binding of Cr and Pb metal ions and the formation of a complex. These functional groups bind to Cr and Pb metal ions during adsorption. Hydrocarbons and silicates are also recorded in the FTIR spectra for all adsorbents. There is a stretching of Si–O–Si and Si–O–Al bonds between the peaks at 950 cm^{-1} and 1000 cm^{-1} , and the vibration of Al–O–Si bonds occurs within the range of 600 to 800 cm^{-1} [27–29]. The band at 908 to 912 cm^{-1} is likely due to the stretching of Al–OH bonds or structures involving Si–O–C or Si–O–Si [30]. The presence of Si and Al in the adsorbent significantly affects the adsorption capacity. Alnajjar et al. [31] studied the silica-alumina composite as an adsorbent for the removal of Metformin from water. They found that the silica and alumina in a compound can provide a high surface area and a high number of strong acidic sites, which affect surface properties (functionality and charge) and are responsible for adsorption [31]. Other researchers also found that Si and Al in magnetic gamma alumina nanoparticles (MGAl-NPs) contribute to the adsorption of heavy metals from synthetic wastewater [32].

4. Conclusions

This study demonstrated that activated sludge obtained from a drinking water treatment plant can be used as an effective adsorbent for removing Cr(III) and Pb(II) ions from aqueous solution. The study revealed that contact time, adsorbent dosage, and pH level significantly affected adsorption capacity. XRF analysis revealed silica, alumina, and iron oxide as the primary structural elements, while SEM pictures revealed surface alterations and aggregation, indicating metal deposition. Peak shifts and the emergence of additional bands at 3401.88 cm^{-1} , 1638.18 cm^{-1} , 1392.88 cm^{-1} (for Cr(III)), and 1397.91 cm^{-1} (for Pb(II)) indicated complex formation, confirming that hydroxyl and carboxyl groups are actively involved in metal binding. Single-metal synthetic solutions were used in this study, while real wastewater contains organic content and a variety of competing ions that could impair performance. Future studies are recommended to assess performance in multi-component systems and in actual industrial effluents, to conduct in-depth modeling to deepen understanding of the adsorption mechanism, and to examine the adsorbent's stability and regeneration potential to facilitate scale-up and long-term application. Overall, this study demonstrates that activated sludge is an accessible, promising, and effective adsorbent for heavy metal removal.

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Conflicts of Interest: The authors declare no conflict of interest.

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