

Use of semi-coke as Pb ion adsorbent from polluted industrial wastewater

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GRAPHICAL ABSTRACT



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ABSTRACT

Due to the increase in water pollution, especially heavy metals, many ways have been proposed to absorb and remove them. So far, various adsorbents have been used to remove pollutants. Semi-coke, a carbonaceous material, is susceptible to use as an adsorbent due to its unique properties, such as good specific surface area and porosity. Therefore, in this study, it was added to a solution containing lead ions after preparing semi-coke particles. The effect of various parameters such as pH, adsorbent dosage, and contact time on the ability of semi-coke to absorb lead ions was investigated. The results showed that semi-coke has a negative surface charge so that it can absorb small ions on its surface by an electrostatic attraction. The optimal pH values, adsorbent dosage, and contact time were obtained by investigating the results as 5, 2.0 g, and 60 min, respectively. Under optimal conditions, semi-coke could absorb 94 % of lead ions from the solution.



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1. Introduction

Following the increasing population and the progress of industries, environmental pollution, especially the pollution of water resources, has increased. The main causes of water pollution are the effluents of various industries, including the automotive industry, chemicals, electronics, and metal industries. Water hardness, heavy metals, and microbial contamination are the three main factors that reduce the usefulness of water (Koohestani, 2019, Ince and Ince, 2017). Heavy metals, including mercury, arsenic, cadmium, nickel, copper, lead, chromium, zinc, vanadium, etc., are important environmental pollutants and threaten the health of living organisms. So far, several methods such as oxidation-reduction processes, precipitation, electrochemical cells, ion exchange, solvent extraction, reverse osmosis, and surface

absorption have been presented and investigated to reduce water pollution and remove heavy metals in water (Arora, 2019, Lin and Juang, 2002). Each of these methods has its details, advantages, and disadvantages. Surface adsorption is a simpler process than other methods. Surface adsorption describes the tendency and attraction of fluid-phase molecules around the adsorbent to accumulate on the solid surface. Therefore, adsorption is a set of interactions between a compound (adsorbate) in the liquid phase and another surface (adsorbent) in the solid phase through weak physical forces (for example, van der Waals forces), chemical interactions (covalent bonds), or electrostatics (Abas *et al.*, 2013, Zamee *et al.*, 2021). In addition to the method used to remove heavy metal pollutants, the type of adsorbent also plays an important role in the purification process. The absorber itself may also cause pollution and destruction of the

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environment. Among the materials used as adsorbents to reduce heavy metals in the effluents of various industries, we can mention coal products and carbon-based materials such as activated carbon, graphite, graphene, or zeolite ore (Zaimee *et al.*, 2021, Xu and Wang, 2017, Hussain *et al.*, 2021).

Activated carbon is known among adsorbents due to its unique characteristics, such as the large surface area, large pore volume (mesopores and micropores), appropriate pore size distribution, surface chemistry, and high adsorption capacity (Nowrouzi *et al.*, 2017). These adsorbents are important in separating odors, colors, and unwanted tastes in domestic and industrial operations, solvent recycling, and air purification, especially in restaurants, food, and chemical industries (Erto *et al.*, 2013, Uzun and Güzel, 2000).

Zeolites constitute a large family of hydrous alumina/silicate minerals. The structural network of zeolite is an aluminosilicate framework that forms an endless three-dimensional network of SiO_4 and Al_2O_3 tetrahedra, which are connected by oxygens so that no non-covalent oxygens are present (Velarde *et al.*, 2023, Koohestani *et al.*, 2019).

As a natural amino polysaccharide, Chitosan has a unique structure and multifunctional properties. The high absorption capacity of chitosan and its derivatives to remove heavy metal ions from water and wastewater can be attributed to the presence of multifunctional amine and hydroxyl groups, high hydrophobicity due to a large number of hydroxyl groups on glucose units, high chemical reactivity of these groups and the flexible structure of the polymer chain (Ahmad *et al.*, 2015, Zhang *et al.*, 2016, Falsafi *et al.*, 2020).

Investigating the absorption mechanism of these materials showed that carbon-based materials have a negative electric charge. When a solution containing pollutants or organic compounds that have positive ions passes through the bed of carbon materials, a large amount of contaminants are absorbed due to the high surface area, high adhesive strength, and electric charge of carbon (Zarei *et al.*, 2022, Abas *et al.*, 2013).

Semi-coke (SC) is also expected to have useful performance as an adsorbent because this material is a carbon product with a high carbon percentage, as well as high resistance and chemical activity (Yao *et al.*, 2022, Guo *et al.*, 2013). SC or lanthan is obtained from burning coal. SC has distinctive features such as high strength, high chemical activity, high electrical resistance, high fixed carbon, developed pore structure, and low ash and sulfur. SC is used in metallurgical, chemical, smelting, and gas industries (Koohestani and Hasanpoor, 2023, Etemadi *et al.*, 2023, Salmani *et al.*, 2023). SC is usually composed of mineral and organic materials with abundant oxygen-containing functional groups ($-\text{OH}$, $\text{C}_2\text{H}_5\text{O}-$, $\text{C}=\text{O}$, etc.) and microporous structures, which can be a potential material for adsorbing various pollutants. However, in practical applications, tar and ash on the surface can limit the absorption of pollutants by SC (Wang *et al.*, 2020, Guo *et al.*, 2013, Li *et al.*, 2024). Of course, with physical and/or chemical activation methods, the surface structure of SC can be modified (Gao *et al.*, 2017). There are a few reports of semi-coke absorption properties for nitric oxide, mercury vapor, Cr^{6+} , and methylene blue (Yang *et al.*, 2015, Huawei *et al.*, 2014).

This work investigates SC's ability to adsorb heavy metal ions in an aqueous solution. Water contaminated with lead ions was used. The experimental data of adsorption were analyzed by changing various parameters such as ion concentration, pH, and contact time.

2. Materials and methods

In this work, SC was obtained as an adsorbent from the Iran Ferrosilicon factory. Its chemical composition includes carbon (84.65%), ash (73.7%), volatile matter (62.7%), and moisture (10%).

Fig. 1 shows a microscopic image of semi-coke particles. Lead nitrate (Arvin Shimi) and hydrochloric acid (Merck) were used.

First, half of the SC was washed with distilled water and ethanol to remove impurities and softeners from the particles' surfaces. Then, the SC particles were dried in an oven. 0.4 g of lead nitrate was added to 100 ml of distilled water to produce contaminated water. After the homogenization of the solution, SC with size $<710\text{ }\mu\text{m}$ was added to it with different weight amounts. After stirring the solution at various times, the lead concentration was determined. This work used an inductively coupled plasma optical emission spectroscopy (ICP-OES, Genesis model, manufactured by Axis Pro). In Table 1, the conditions for performing various tests are presented. Then, the effect of important parameters such as pH, adsorbent amount, and contact time will be investigated.

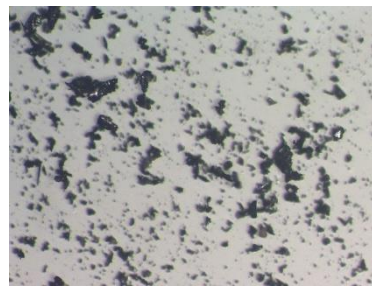


Fig. 1. Microscopic image of semi-coke particles.

Table 1. Specifications of the performed tests and their naming.

Test code: pH-time-weight	pH	Contact time, min	Weight of adsorbent, g
T:7-120-1	7	120	1.0
T:3-60-1.5	3	60	1.5
T:5-60-1.5	5	60	1.5
T:5-40-1.5	5	40	1.5
T:5-80-1.5	5	80	1.5
T:5-60-2	5	60	2.0
T:5-60-2.5	5	60	2.5

SC phase studies were performed by X-ray diffraction (XRD) using a Bruker D8 Advance diffractometer with $\text{Cu-K}\alpha$ radiation ($\lambda=0.1544\text{ nm}$). SC's Zeta potential and particle size distribution were determined using dynamic light scattering (DLS) with a Malvern Nano-ZS90 analyzer. Fourier transform infrared (FTIR) analysis was performed using a Shimadzu FTIR 8400S spectrometer to obtain information on the surface properties of the SC.

3. Results and discussion

Fig. 2 shows the XRD pattern of SC. Carbon peaks and some amorphous structures can be seen in the SC. Two peaks at 26° and 43° are the main carbon peaks and correspond to (002) and (100) planes, respectively. The (002) peak shows the parallel orientation of aromatic carbon layers in microcrystals. The peak (100) represents the size of an aromatic carbon sheet (Kumar *et al.*, 2019, Lv *et al.*, 2021). Impurity peaks are also observed, but there is a large amount of carbon in the SC.

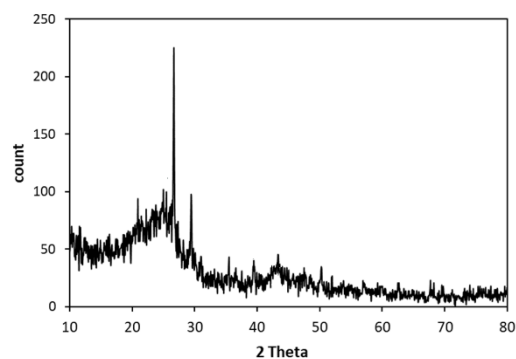


Fig. 2. XRD pattern of semi-coke.

Fig. 3 and Table 2 present the results of the FTIR analysis of semi-coke. The peaks in the range of $400\text{--}700\text{ cm}^{-1}$ (791 cm^{-1} , 685 cm^{-1} , 528 cm^{-1} , and 472 cm^{-1}) are related to Si-O , Si-O-Si , and Si-O-Al bonds (Ding *et al.*, 2022, Wang *et al.*, 2021).

In addition, the characteristic peaks at 2920.03 cm^{-1} and 2852.52 cm^{-1} appear to be symmetric and asymmetric stretching vibration towards $-\text{CH}_2$ and $-\text{CH}_3$, the characteristic peak at 1461.94 cm^{-1} corresponding to $-\text{OH}$ or asymmetric tension $-\text{COO}$ and tension $-\text{COO}^-$ (Zhang *et al.*, 2023, Wang *et al.*, 2021, Ma *et al.*, 2022).

Aliphatic stretching vibrations are specifically assigned to symmetric and asymmetric CH_2 stretching vibrations (Ding *et al.*, 2022).

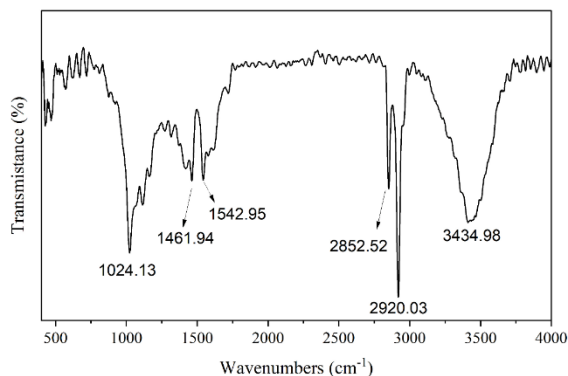


Fig. 3. FTIR spectrum of semi-coke.

Table 2. FTIR of semi-coke.

Wave number/cm ⁻¹	Assignment
700–400	Si-O, Si-O-Si, and Si-O-Al bending vibrations
1024.13	C-O stretch
1461.94	Si-O-Si stretching vibrations
1542.95	C=C stretch
2852.52	Asymmetrical CH ₃ bending vibration
2920.03	C=OOH stretch
3434.98	Symmetrical CH ₂ stretching vibrations
	Asymmetrical CH ₂ stretching vibrations
	C-H stretch

Table 2 presents the amount of lead absorption in different tests. The test T:7-120-1, done at pH=7, absorbed only 13% of lead. At pH=7, the adsorbent did not have good ability, but it shows that SC can absorb lead. Of course, in such conditions, the ability of activated carbon to absorb cadmium and lead is reported to be about 65% and 83%, respectively (Dowlatshahi *et al.*, 2014).

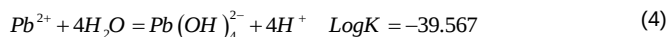
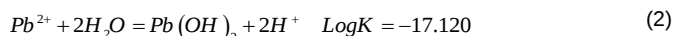
Therefore, since factors can improve the absorption ability of SC, the effect of the parameters of the contact time between the adsorbent and the solution, the amount of adsorbent, and the pH was studied.

Table 3. Amount of lead absorption by SC in different tests.

Test number	Test code	Absorption of Pb, %
1	T:7-120-1	13
2	T:3-60-1.5	5
3	T:5-60-1.5	87
4	T:5-40-1.5	63
5	T:5-80-1.5	76
6	T:5-60-2	94
7	T:5-60-2.5	82

Test T:3-60-1.5, where the adsorbent amount was increased to 1.5 g and the contact time and pH were reduced compared to the first test, only 5% pollutant removal was achieved, which is very low. Therefore, in addition to increasing the amount of adsorbent, the effect of two other parameters has been more intense, which has reduced the amount of lead absorption.

T:5-60-1.5, the pH value has increased to 5, leading to 87% of Pb absorption. Therefore, pH is a very effective parameter. In a similar study on the process of Pb absorption by graphene nanocomposite, the amount of absorption was reported to be 92% (Kumar *et al.*, 2019). The optimal pH value is 5 because the adsorbent particles dissolve in the solution when the pH is less than 4. Therefore, the absorption percentage will be lower (Test 2). At a pH higher than 6, the metal ions in the solution have a neutral surface charge, and the adsorbent cannot absorb them (Test 1). Therefore, the best pH can be considered 5. pH is an important factor affecting the adsorption of metal species in aqueous solutions and the activity of functional groups. This factor facilitates understanding the effect of pyrolysis conditions on the charges on the SC surface and the mechanisms involved in the adsorption process (Silva *et al.*, 2022, Tran *et al.*, 2020). In various studies, the distribution of lead species has been investigated as a function of pH. Their information is obtained based on the following reaction equilibrium constants (Silva *et al.*, 2022, Yang *et al.*, 2011, Xu *et al.*, 2008):



It is observed that at acidic-neutral pH, lead exists mainly in the ionic form Pb^{2+} . At low pH, there is competition between Pb^{2+} and H^+ for adsorption sites (Silva *et al.*, 2022, Varadwaj *et al.*, 2017, Yang *et al.*, 2011). The increase in Pb^{2+} adsorption on the SC can probably be attributed to the hydrolysis of Pb^{2+} in aqueous solution (Xu *et al.*, 2008).

Wang *et al.* (Wang *et al.*, 2023) showed that due to the functional groups of blue coke, the removal rate of Cr^{6+} by it decreases sharply with increasing pH, and the ideal pH is 2. Cheng *et al.* (Cheng *et al.*, 2023) also reported that with increasing pH, the adsorption capacity of Cu^{2+} by biotransformed-lignit (BL) gradually increased and reached the maximum at pH=6 due to the easier replacement of hydrogen ions in the active groups of BL.

Zhang *et al.* (Zhang *et al.*, 2018), in a similar study that investigated the effect of pH on the absorption of Cr^{6+} by SC, showed that the highest efficiency was obtained at pH=5. In a study, Zhao *et al.* (Zhao *et al.*, 2022) observed that the adsorption of Cr^{6+} on the SC surface did not change with increasing pH, which proves that electrostatic interaction is not important in Cr^{6+} absorption.

Increasing the adsorbent reduces the free space, and even particles stick together. Therefore, the absorption efficiency will decrease with increased apparent viscosity (Zhao *et al.*, 2022). Small micro- and mesopores are in the SC, where lead atoms enter these adsorbent pores. Therefore, pore absorption plays an important role in this process. Based on kinetic studies, the semi-coke chemisorption process for Pb^{2+} conforms to a pseudo-second-order model (Wang *et al.*, 2020).

In the T:5-40-1.5, where the contact time has been reduced to 40 min, the absorption rate was 63%. Therefore, the contact time has been an impact parameter that has caused the absorption efficiency to decrease from 83% to 63%. Hence, in the T:5-80-1.5, the contact time was increased to 80 min. However, the absorption value was reported as 76%, which shows that increasing the contact time has reduced the efficiency. Therefore, the best call duration is 60 min. In the initial times of contact, there are empty places on the surface of the adsorbent for the absorption of metal ions. But over time, these places are occupied, and the contact between the adsorbent and the contaminated solution continues. Hence, metal rejection from the adsorbent surface occurs, leading to a decrease in the performance of the absorption process (Kumar *et al.*, 2019, Silva *et al.*, 2022).

Fig. 4 shows SC particles' zeta potential and size distribution at pH=5. As can be seen, the zeta potential of SC is negative (about -20 mV). Therefore, the electrostatic attraction between the positive lead ions and the negative charge of the SC surface is also an essential factor in the absorption process (Zhao *et al.*, 2022). Based on the study by Guo *et al.* (Guo *et al.*, 2013) on the functional groups C=O, -NH₂, etc. on the surface of SC and their role in the adsorption reaction, and according to the SC FTIR pattern in Fig. 3, it can be stated that the functional groups participate in the adsorption process. Meanwhile, according to Fig. 4, the average size of SC particles is about 304 nm, and the Polydispersity index (PDI) =0.495 shows that the particle size distribution is relatively wide and in the range of 100 to 500 nm.

Test T:5-60-2, which has a higher amount of adsorbent and showed an absorption rate of 94 %. This result, which was the best case, indicates that SC, like other carbon-based adsorbents, can absorb heavy metals. Absorption of physical penetration plays an important role in absorption processes. Of course, chemical absorption also significantly contributes to carbon materials' absorption capacity (Zhao *et al.*, 2022, Mao *et al.*, 2020, Dong *et al.*, 2019). Destruction of the dense structure of SC and turning it into a porous structure with a high specific surface can increase its absorption ability (Zhao *et al.*, 2022). Wang, *et al.* (Wang *et al.*, 2020) obtained the removal efficiency of Pb^{2+} with a concentration of 200 mg/L by semi-coke at pH 5.5 to be 80.23%. They stated that the difference in the removal ability of heavy metals by semi-coke is due to the hydration radius of metals and the hydration energy of cations.

In the continuation of the test work, T:5-60-2.5 was investigated, and the pollutant absorption was 82%. It shows that SC has a high absorption capacity, but it was unfavorably compared to the previous sample.

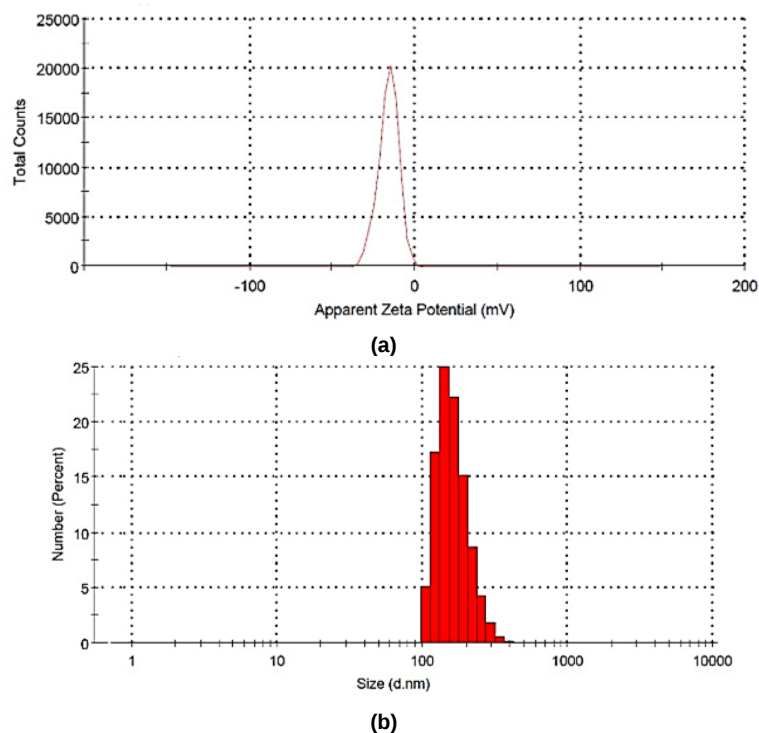


Fig. 4. (a) Zeta potential, and (b) size distribution of SC particles at pH=5.

4. Conclusions

This research proposed semi-coke as an adsorbent to remove heavy metals from water due to its unique properties. Based on this, the performance of semi-coke in absorbing lead ions from water was evaluated. Due to the negative surface charge of semi-coke, it can adsorb positively charged lead ions through surface functional groups. The results showed that at pH=5, the adsorption efficiency of lead ions is about 94%. Considering the effectiveness of the parameters of contact time and adsorbent amount, their optimum state was obtained as 60 min and 2.0 g. Therefore, by investigating the effective parameters, it is possible to use semi-coke, a carbon material that causes fewer biocompatibility problems, to absorb metal ions from water.

Author Contributions

Hassan Koohestani: Conceptualization, investigation, methodology, review, and editing.

Abolfazl Gohariyan Bajestani: methodology, analysis, and writing-original draft

Conflict of Interest

The authors declare no competing interests and non-financial competing interests.

Data Availability Statement

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

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