Strontium-doped hydroxyapatite as adsorbent effectively to remove lead ions from water

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Abstract
In this study, a strontium-doped hydroxyapatite (Sr-HAP) was synthesized by the solgel method, which was used as adsorbent to remove lead ions (Pb2+) from water. The results showed that the adsorption capacities of the Sr-HAP were obviously higher than those of the HAP, the adsorption capacities of which for Pb2+ reached 651.175 mg/g. The proper increasement in the dosage of adsorbent was beneficial to the removal of Pb2+ by Sr-HAP. Meanwhile Sr-HAP had a wide applicable pH range for Pb2+. And the increasement in temperature could increase the adsorption capacity of Sr-HAP for Pb2+ to a certain extent. The Langmuir model was used to fit the isotherm adsorption process of Sr-HAP to Pb2+ in water. Compared with HAP, the specific surface area of Sr-HAP has increased by 11.1%, and the pore size distribution of Sr-HAP tended to be smaller and more uniform. Hence, Sr-HAP could be used as an ideal adsorbent to remove Pb2+ in wastewater.

Keywords Hydroxyapatite · Adsorption · Strontium-doped · Lead ions · Mechanism

Introduction
Heavy metals pollution was known to cannot undergo biodegradation, resulting in adverse effects on the environment and biological systems (Imron et al. 2019; Purwanti et al. 2020). Lead was one of the heavy metals, which was the second most toxic metal and often found in the environment, and then led to no safe level of lead in drinking water (Adeyemi et al. 2021; Kim and Lee 2017; Du et al. 2017). It was acutely toxic to human body when consumed in high concentrations inside cells (Nwaehiri et al. 2020). Under environmental lead exposure, the elevated lead blood levels could make the Brain system for the permanent and irreversible damage for human, especially the young children (Cecil et al. 2008). At present, the existing removal technologies for lead-contaminated wastewater mainly include chemical precipitation method (Ma et al. 2019), ion exchange method (Badmus et al. 2021), membrane filtration method (Mishra 2014), flotation method (Jia et al. 2022), adsorption method (Hashem et al. 2020), biosorption method (Rangabhashiyam and Balasubramanian 2019) and electrochemical method (Li et al. 2015). Among them, the adsorption method due to its simple operation process, environmental friendliness and high removal efficiency was an important method to remove lead ions (Pb2+) from wastewater.

Hence, much more attention had been paid to the adsorbent selection. Activated carbon (AC) (Kongsuwan et al. 2009), zeolite (Chong et al. 2013), chitosan (Paulino et al. 2007), ion exchange resins (Dong et al. 2010), industrial solid waste (Yong 2018), nanomaterials (Li et al. 2020), hydroxyapatite (HAP) (Aklil et al. 2004; Wen et al. 2013; Fernane et al. 2013; Lei et al. 2015; Wang et al. 2017; Foroutan et al. 2020, 2021), etc., were widely used as the adsorption materials. Among them, the HAP with the advantage of large surface area, low cost and high stability was used as the adsorbent for lead removal efficiently and greatly promoted practical applications. Wang et al. (Wang et al. 2017) studied the
magnetic hydroxyapatite-mixed oxidation of multi-walled carbon nanotubes (mHAP-oMWCNTs) to remove Pb²⁺ in water, and the results showed that mHAP-oMWCNTs showed better performance than mHAP, mMWCNT and HAP-oMWCNTs. Aklil et al. (2004) used calcined hydroxyapatite to adsorb Pb²⁺, Zn²⁺ and Cu²⁺ and found that at pH 5, the adsorption capacity of HAP on Pb²⁺, Zn²⁺ and Cu²⁺ was 85.6, 29.8 and 20.6 mg/L, respectively. Lei et al. (2015) prepared a hydroxyapatite/chitosan (HAP/CS) porous material with a pore size of 100–300 mm to adsorb Pb²⁺, and its maximum adsorption capacity was 264.42 mg/g. Meanwhile, adding extra elements could improve the performance of hydroxyapatite. Tang et al. (Wen et al. 2013) prepared Si-CHAP by hydrothermally doping silicon carbon into hydroxyapatite. The results showed Si-CHAP with a large specific surface area of 323.25 m²/g, thereby enhancing the adsorption capacity.

Introducing Sr²⁺ with a larger ionic radius into the crystal structure of hydroxyapatite to replace part of Ca²⁺, making the lattice larger, which is more conducive to the substitution of Pb²⁺, thereby improving the removal ability of hydroxyapatite to Pb²⁺. Hence, in this work, a strontium-doped hydroxyapatite (Sr-HAP) was synthesized by the solgel method. The Pb²⁺ adsorption experiment was carried out using the prepared material. The adsorption behavior and influence of Sr-HAP on Pb²⁺ were discussed under different adsorption conditions. The adsorption kinetic equation and thermodynamic parameters were calculated, and the recycling rate of Sr-HAP was explored. Meanwhile, the Sr-HAP was investigated by the various analytical techniques, such as X-ray diffractometer, Fourier transform infrared, scanning electron microscope, Brunauer–Emmett–Teller, zeta potential analyzer. Finally, the adsorption mechanism of Sr-HAP on Pb²⁺ was explained.

**Materials and methods**

**Adsorbent preparation and characterization**

**Preparation**

The Sr-HAP sample with $x = Sr/(Ca + Sr)$ molar ratio of 0.5 was prepared by the solgel method. 1 mol/L Sr(NO₃)₂, Ca(NO₃)₂·4H₂O and (NH₄)₂HPO₄ solution were diluted to 0.2 mol/L, respectively. The certain volume of Sr(NO₃)₂·6H₂O solution, Ca(NO₃)₂·4H₂O solution and (NH₄)₂HPO₄ solution were mixed, in which the (Sr + Ca)/P molar ratio was fixed at 1.67. Meanwhile, ammonia water was added quickly into the above mixture to adjust the pH within 10–11. Next, the mixed solution was stirred quickly for 1–2 min, then the magnetic stirring was kept for 30 min, and the obtained solution was transferred in the water bath (aging) at 50 °C for 24 h. After reaching the reaction time, the pH of the supernatant was measured by natural cooling and the solgel was centrifuged (4000 r/min, 5 min) and washed with ultrapure water for three times (to neutral) and then washed with absolute ethanol. After drying at 80 °C for 24 h, strontium-doped hydroxyapatite (Sr-HAP) was prepared after grinding finely (sieved through 100 mesh, and stored in the dry environment).

**Characterization**

The specific surface area and pore size distribution curve of the sample were measured by the NOVAe1000 (Conta Instruments, USA) specific surface area and pore size distribution analyzer. X-ray diffractometer (XRD) analysis of the synthesized sample was carried out using XPert PRO. The phases of the sample were determined by comparison with the standard PDF card using “PAN analytical XPert High-Score”, and the X-ray diffraction patterns before and after the sample adsorbed Pb(II) were recorded. Fourier transform infrared absorption spectrometer (FT-IR, Thermo Nexus 470FT-IR) was used to determine the functional groups contained in the synthesized Sr-HAP and verify the structure of the sample. The morphology and surface chemical composition of the sample were analyzed by a scanning electron microscope (SEM, JSM-7900F) and an electron-dispersive X-ray analyzer (EDS). Zeta potential analyzer (Zetasizer Nano ZS90) was used to measure the zero-point potential.

**Adsorption experiments**

Pb²⁺ solution was prepared by analytically pure lead nitrate, nitric acid and ultrapure water. The 0.2% nitric acid solution was used to dilute all samples. The batch experiment was carried out in a set of 100-mL polyethylene centrifuge tubes, and KOH or HNO₃ solution was used as pH regulator. The Sr-HAP sample was added into the certain amount of Pb²⁺ solution which was prepared by lead nitrate and then the mixed solution was placed in a constant-temperature water bath shaker at 180 r/min. Oscillating at a constant temperature for 24 h, the temperature was set as 25 °C. After the sample was filtered with a syringe filter (0.22-μm filter membrane), the residual Pb²⁺ concentration in the solution was measured.

The influence of different initial concentrations of Pb²⁺ solutions (100, 200 and 300 mg/L) and pH 5.0 (±0.05) solutions and different dosages of Sr-HAP (0.01–0.1 g, <100 mesh) on adsorption was investigated at 25 °C by using nitric acid and sodium hydroxide (0.1 mol/L) solution to adjust the initial pH value of Pb²⁺ solution to study the effect of pH on adsorption. 50 ml containing Pb²⁺ wastewater with an initial pH of 5.0 was prepared, which had different concentrations.
(100, 150, 200, 250, 300, 400, 500, 600, 700, 800, 900 and 1000 mg/L). Then, 40 mg of Sr-HAP and HAP was added to the Pb²⁺ solution to determine the effect of the initial concentration of the solution on the adsorption at the constant temperature of 25 °C, 35 °C and 45 °C, respectively. Meanwhile, the isothermal adsorption and thermodynamic characteristics were analyzed. Water samples were collected at different times (5, 10, 15, 30, 60, 120, 240, 300, 360, 420, 480, 540, 720, 1080 and 1440 min) to study the effect of adsorption time on the adsorption. 40 mg of Sr-HAP solid with particle size less than 100 meshes was added to a mixed solution, which consisted of 50 mL of Pb²⁺ solution with an initial concentration of 100 mg/L and different concentrations (0, 10, 20, 40, 60, 80, 100, 150 and 200 mg/L) of Mg²⁺, Ca²⁺ and EDTA aqueous solutions, or Pb²⁺ solution (50 mL, 100 mg/L) and different concentrations (0, 1, 2, 5, 8, 10, 15, 20, 50 and 100 mg/L) of fulvic acid (FA) or black humic acid (HA) solution to study the influence of interfering substances on adsorption. Inductively coupled plasma emission spectrometer (Opetima 7000DV) was used to determine the residual Pb²⁺ concentration in the solution.

Desorption experiments

Sr-HAP samples containing Pb²⁺ were desorbed with different eluents. The adsorbent saturated under the optimal adsorption conditions was washed with deionized water for 3~5 times and dried at 80 °C. 0.1 mol/L desorption solution (NaOH, HCl, NaCl), H₂O, TCLP (pH = 2.55 and 4.93), Ca(NO₃)₂ (0.001, 0.005 and 0.01 M), Sr(NO₃)₂ (0.001, 0.005 and 0.01 M) were oscillated under the same conditions, then the mass concentration of the filtrate was measured after filtration, and the desorption rate was calculated.

Results and discussion

Characterization of the adsorbent

The X-ray diffraction (XRD) patterns of Sr-HAP with different adsorption concentrations of Pb²⁺ are shown in Fig. 1(A). It could be seen that after Pb²⁺ adsorption, the crystal structure of Sr-HAP changed significantly, and the position of the characteristic peaks changed too. The intensity of the main characteristic peaks changed with the increase in Pb²⁺ concentration, but it still belonged to the hexagonal crystal structure of apatite, and the space group was P6₃/m. Figure 1(B) shows the angular deviation of the characteristic peaks after magnification. It could be seen that the characteristic peak of the (211) crystal plane gradually divided into two different positions (112 crystal plane and 300 crystal plane) as the concentration of adsorbed Pb²⁺ increased. The formation of a new peak at 2Θ of 30.1°~30.5° corresponded to the reference substance Pb₁₀(PO₄)₆(OH)₂ (HPY) or PbₓCa₅₋ₓ(PO₄)₃OH solid solution with lower calcium content. Because the peaks at 30.3° and 31.8° were determined as the diffraction characteristic signals of HPY and HAP in the solid, the ratio of these two peaks could semi-quantitate the conversion of HAP to HPY. The ionic radius of Pb²⁺ was 1.20 Å, and it was larger than that of Sr²⁺ (1.12 Å). Compared with Ca²⁺ (0.99 Å), it was difficult for Pb²⁺ to enter the Sr-HAP crystal to replace Ca²⁺.
and Sr$_2^+$. Entering the HAP lattice through the substitution of calcium ions was difficult for lead ions. Therefore, the lattice-substituted Pb might come from the subsequent diffusion of surface-adsorbed Pb to the particle core. It indicated that the dissolution/precipitation effect might exist in the removal of Pb$_2^+$ by Sr-HAP.

The Sr-HAP solid phase adsorbed with different concentrations of Pb$_2^+$ was analyzed by infrared spectroscopy to compare the changes of functional groups (Fig. 2). The typical characteristic peaks of Sr-HAP with different adsorption concentrations of Pb$_2^+$ could be seen, which was almost identical to that of pristine Sr-HAP indicating that its functional group structure did not change significantly. With the increase in the Pb$_2^+$ adsorption concentration, the stretching vibration peak intensity of PO$_4^{3-}$ at the wavelengths of 1033.31 and 561.08 cm$^{-1}$ changed significantly, indicating that PO$_4^{3-}$ participated in the chemical reaction in the adsorption process. Meanwhile, the morphology of Sr-HAP was analyzed by the scanning electron microscope (SEM). It could be seen from Fig. 3 that the morphology of the material presented scattered rod-like or needle-like structured crystals, with the uneven particle morphology and the large pores between the crystal grains.

Figure 4(A) shows the typical N$_2$ adsorption–desorption isothermal curves of the synthesized HAP and Sr-HAP materials, respectively. As shown in Fig. 4(A), Sr-HAP and HAP exhibited type IV isotherms with H3 hysteresis loop; according to IUPAC’s empirical classification, the material was composed of mesoporous structure. It is generally believed that the shape of the
Hysteresis loop is related to the texture of the porous material (for example, pore size distribution, connectivity and geometry), and hysteresis loop material has wedge-shaped pores with open ends (H3-type isotherm did not show any limited adsorption under high P/Po, which could be observed in non-rigid aggregates of plate-like particles) (Alothman 2012), which was consistent with the images of scanning electron microscope. Compared with HAP (102.314 m²/g), the specific surface area of Sr-HAP (115.121 m²/g) increased by 11.1%. Figure 4(B) shows the pore size distribution of HAP and Sr-HAP. It could be seen that the pore size of the material was mainly distributed in the range of 2–50 nm, which belonged to the mesoporous materials. The pore size distribution of Sr-HAP was smaller than that of HAP, and the pore size was more uniform. It showed that the incorporation of strontium caused the material lattice defects and the reduction of the particle size, and the pores between the particles were also reduced.

From Fig. S1, it could be seen that the zero point electricity of Sr-HAP was pHpzc = 8.40, and the zero electricity point of HAP was pHpzc = 6.92. Due to the special structure of apatite (M(PO₄)₃OH), the material had a certain buffering capacity, which could maintain its own acidity and alkalinity in the acid or alkaline environment, and released OH⁻ or PO₄³⁻. When the pH value of the solution was lower than the zero electric point, Me²⁺ would electrostatically repulse the positive charge on the surface of the material. When it was higher than its zero electric point, Me⁴⁺ would electrostatically attract the positive charge on the surface of the material. For Me²⁺, when the pH value of the solution was higher than the zero electric point of Sr-HAP, Me²⁺ was easy to hydrolyze. At this time, the removal of Me²⁺ was caused by its own characteristics. Heavy metal wastewater is generally weak acid or neutral, so it is more reasonable for removal heavy metal ions to choose the material the pH value of which is lower than the zero electric point.

**The influence of adsorption conditions**

**Influence of adsorbent dosage**

For Pb²⁺-containing solutions of different concentrations, the dosage of adsorbent has a certain effect on the removal of Pb²⁺. It could be seen from Fig. 5 that the removal rate of Sr-HAP adsorbent for solutions with different initial Pb²⁺ concentrations (100, 200 and 300 mg/L) increased with the increasement in the dosage. When the dosage of Sr-HAP was 40 mg / 50 mL, the removal rate curves of different initial Pb²⁺ concentrations gradually tended to be flat. The unit adsorption capacity of Sr-HAP on Pb²⁺ gradually decreased with the increase in the adsorbent dosage. The increasement in available active sites on the adsorbent surface might be attributed to the increase in the dosage of Sr-HAP, while the amount of Pb²⁺ was limited, resulting in the unit adsorption capacity of Sr-HAP to Pb²⁺ gradually decreased. When the initial concentration of Pb²⁺ was 100 mg/L, the adsorption capacity of Pb²⁺ decreased from 403.481 mg/g to 42.377 mg/g with the adsorbent dose increasing from 10 to 100 mg in 50 mL solution. At the
same time, the corresponding Pb\(^{2+}\) removal rate increased from 99.88% to 99.91%. For the initial Pb\(^{2+}\) concentration of 200 mg/L in a 50 mL solution, the adsorption capacity of Pb\(^{2+}\) increased from 330.686 mg/g to 466.733 mg/g with the adsorbent dosage increasing from 10 to 20 mg, and then the adsorption capacity of Pb\(^{2+}\) decreased from 466.733 mg/g to 94.953 mg/g with the adsorbent dosage increasing from 0.02 g increased to 0.10 g. The corresponding Pb\(^{2+}\) removal rate had risen from 35.51% to 99.95%. When the initial concentration of Pb\(^{2+}\) was 300 mg/L and the solution volume was 50 mL, the dosage of adsorbent increased from 10 to 40 mg, and the adsorption capacity of Pb\(^{2+}\) increased from 298.559 mg/g to 350.052 mg/g. The dosage increased from 40 to 100 mg, and the adsorption capacity of Pb\(^{2+}\) decreased from 350.052 mg/g to 142.641 mg/g. Meanwhile, the removal rate of Pb\(^{2+}\) by Sr-HAP gradually increased from 22.63% to 100.00%. When the dosage of Sr-HAP was 40 mg/50 mL, the removal rate of Pb\(^{2+}\) was above 98.00% at different concentrations (100, 200 and 300 mg/L), and the adsorption capacity was relatively large, so 40 mg/50 mL was used as the optimal adsorbent dosage for further experiments.

**Influence of the initial pH value of the solution**

The pH value of the solution was one of the important parameters to control the metal ion removal mechanism (Hao et al. 2017). Figure 6(A) shows that when the pH value of the solution changed from 1.0 to 2.0, the removal rate increased, and the adsorption capacity also increased. When the pH value changed from 2.0 to 5.0, the removal rate remained basically unchanged and the adsorption capacity also tended to be stable. However, when the pH value changed from 5.0 to 8.0, the removal rate did not change much, but the adsorption capacity gradually decreased. The pH value of the solution system not only affected the existence of Pb\(^{2+}\) in the water, but also directly affected the surface charge point of the adsorbent, the pore structure and chemical properties of the material surface (Yang et al. 2013). Pb\(^{2+}\) in the aqueous solution would exist in different forms under different pH conditions. When the pH exceeded 8.0, the lead hydroxide \((\text{Pb(OH)}_2)\) \(K_{sp}[\text{Pb(OH)}_2] = 1.2 \times 10^{-15}\) gradually formed. A part of Pb\(^{2+}\) in the aqueous solution could be removed by precipitation, which added some interference to the removal of Pb\(^{2+}\). Visual MINTEQ v3.0 software was used to simulate the existence of Pb\(^{2+}\) with pH changed at 25 °C and the concentration of 300 mg/L (Bai et al. 2020; Li et al. 2019). As shown in Fig. 6(B), the formation of Pb\(^{2+}\) under different pH conditions included Pb\(^{2+}\), Pb(OH)\(_{2}\)(aq), Pb(OH)\(^{+}\), Pb\(_{2}\)(OH)\(^{3+}\), Pb\(_{3}\)(OH)\(_{4}\)\(^{2+}\), Pb\(_{4}\)(OH)\(_{4}\)\(^{4+}\), Pb(OH)\(^{-}\) and so on. When the pH was less than 6.0, the lead element in the solution mainly existed in the form of Pb\(^{2+}\) ions. In the pH range of 6.0 to 9.0, the concentration of Pb\(^{2+}\) decreased rapidly, and the positively charged Pb(OH)\(^{+}\), Pb\(_{2}\)(OH)\(^{3+}\), Pb\(_{3}\)(OH)\(_{4}\)\(^{2+}\) and Pb\(_{4}\)(OH)\(_{4}\)\(^{4+}\) increased gradually. Hydrolysis products gradually appeared, while the neutral Pb(OH)\(_{2}\) began to precipitate at pH 8.0 and became one of the main existing forms at pH 11. Therefore, the pH value of solution should be controlled within the range of 1.0 to 8.0 to eliminate the influence of Pb\(^{2+}\) precipitation.

In the lower initial pH range (lower than pHPZC), protons were consumed by the protonation of the ≡P-O\(^{-}\), ≡Ca-OH\(^{0}\) and ≡Sr-OH\(^{0}\) groups on the surface, resulting in the increase in the final pH of the solution. In the acidic solution, the surface of Sr-HAP was dominated by positively charged ≡Ca-OH\(^{2+}\), ≡Sr-OH\(^{2+}\) and neutral ≡P-OH\(^{0}\) sites, so that the surface charges of Sr-HAP were positive in this pH range. On the other hand, the final pH decrease occurred in the range of higher initial pH (higher than pHPZC), due to the consumption of ≡Ca-OH\(^{2+}\), ≡Sr-OH\(^{2+}\) and

![](image.png)

**Fig. 6** The effect of the initial pH value on the adsorption of Pb\(^{2+}\) by Sr-HAP (A) and the formation of Pb\(^{2+}\) under different pH (B)
equiv P-OH positions by OH\textsuperscript{−} through surface deprotonation point. Therefore, the neutral equiv Ca-OH\textsuperscript{0}, equiv Sr-OH\textsuperscript{0} and the negatively charged equiv P-O\textsuperscript{−} groups were dominant in the alkaline solution so that the surface of Sr-HAP was negatively charged in the alkaline solution and could adsorb positively charged hydrolyzed substances (such as Pb(OH)\textsuperscript{+}, Pb\textsubscript{2}(OH)\textsubscript{4}\textsuperscript{2+}, Pb\textsubscript{3}(OH)\textsubscript{4}\textsuperscript{4+}, etc.), which was beneficial to the overall removal process. In the removal process of Pb\textsuperscript{2+} by Sr-HAP, for the Pb\textsuperscript{2+}-containing wastewater with the concentration of 100, 200 and 300 mg/L, when the pH value from 2.0 to 6.0, the removal rate was above 97.51% and the adsorption capacity was 100.177, 209.881 and 301.267 mg/g, respectively. It indicated that Sr-HAP was suitable for removing Pb\textsuperscript{2+} in neutral or weakly acidic aqueous solutions, which was beneficial to the application of Sr-HAP to the actual heavy metal wastewater treatment. In this experiment, pH 5.0 was selected for the next experiment, which was consistent with the pH value in the literature (Yan et al. 2014; Zhu et al. 2018).

**Influence of the initial concentration of the solution**

It could be seen from Fig. 7 that the initial concentration was the main factor affecting the removal effect of Pb\textsuperscript{2+}. The overall trend of Pb\textsuperscript{2+} removal at different temperatures was similar. After the initial concentration of Pb\textsuperscript{2+} was increased to 300 mg/L, the removal rate decreased with the increase in the initial concentration of Pb\textsuperscript{2+}, and the adsorption capacity increased with the increase in the initial concentration of Pb\textsuperscript{2+}. When the initial concentration of Pb\textsuperscript{2+} increased from 100 mg/L to 800 mg/L, the removal amount of Pb\textsuperscript{2+} increased from 128.779, 128.481 and 128.474 mg/g to 651.175, 673.806 and 695.223 mg/g, at 25, 35 and 45 °C, respectively. Correspondingly, the removal rate dropped from 99.98%, 100.00% and 99.99% to 65.89%, 68.52% and 71.05%, respectively. The reason why the removal amount of Pb\textsuperscript{2+} by Sr-HAP increased with the increase in the initial concentration by Sr-HAP might be that when the dosage of Sr-HAP was determined, the initial concentration of Pb\textsuperscript{2+} gradually increased and the driving force of the mass transfer of the solution increased the passing rate of Pb\textsuperscript{2+} from the bulk solution to the particle surface, more and more Pb\textsuperscript{2+} accumulated on the surface active sites of Sr-HAP. The decrease in removal rate was attributed to the increase in the initial Pb\textsuperscript{2+} concentration, and the active sites on the surface of the adsorbent were fully occupied. The adsorption was close to saturation, and the removal rate also drops (Chao et al. 2017). It could also be concluded from Fig. 7 that temperature would affect the degree and rate of adsorption. As the temperature increased, Pb\textsuperscript{2+} rapidly diffused and accelerated through the surface of the Sr-HAP particles, and the adsorption capacity and removal rate increased. This indicated that appropriate heating could promote the Pb\textsuperscript{2+} and the removal process might be an endothermic process (Wei et al. 2015).

**Influence of adsorption time**

It could be seen from Fig. 8 that the amount of Pb\textsuperscript{2+} adsorbed by Sr-HAP gradually increased with the increase in adsorption time. In the initial stage of adsorption, Pb\textsuperscript{2+} quickly occupied the active sites on the surface of Sr-HAP, and the Pb\textsuperscript{2+} concentration difference at the solid/liquid interface was large, the solution mass transfer driving force was relatively large, and it was easy to overcome the mass transfer resistance of Pb\textsuperscript{2+} at the solid/liquid interface, thereby accelerating the adsorption rate. With the prolonging of adsorption time, the active sites on the surface of Sr-HAP had been fully occupied by Pb\textsuperscript{2+}, the Pb\textsuperscript{2+} concentration difference at the solid/liquid interface
gradually decreased, the adsorption rate decreased, and the adsorption amount gradually became flat. At about 120 min, the adsorption basically reached equilibrium. At this time, the maximum adsorption capacity of Sr-HAP at different temperatures was 587.077 mg/g (25 °C), 593.863 mg/g (35 °C) and 598.138 mg/g (45 °C). The increasement in temperature could increase the adsorption capacity of Sr-HAP for Pb²⁺, indicating that the adsorption of Pb²⁺ by Sr-HAP was an endothermic process, but it could not shorten the time to reach the equilibrium of the reaction. Meanwhile, the maximum adsorption capacity of HAP at 25 °C was 538.214 mg/g, which lower than Sr-HAP at 25 °C (587.077 mg/g), indicating that introducing Sr²⁺ into HAP could improve the removal ability of hydroxyapatite to Pb²⁺.

**Influence of interfering substances**

It could be seen from Fig. S3 that the higher the EDTA concentration, the lower the removal rate of Pb²⁺ by Sr-HAP. The calculation of EDTA by Visual MINTEQ v3.0 software showed that when pH 5.0, EDTA mainly appeared in the form of H₂EDTA²⁻, which could react with Pb²⁺ at a molar ratio of 1:1 to form negatively charged Pb(II)EDTA²⁻. Lead element existed in the solution in the form of Pb²⁺ and Pb(II) EDTA²⁻ complexes. At lower pH, the adsorption of Me(II) EDTA²⁻ complexes could be achieved on the -SOH site of the adsorbent through surface coordination reactions. Formula (1) to explain:

\[
\text{SOH} + \text{H}^+ + \text{Pb(II)EDTA}^{2-} \rightarrow \text{SOH}^+ + \text{Pb(II)EDTA}^{2-}
\]  

(1)

Lead element was adsorbed on Sr-HAP in the form of Pb²⁺ and Pb(II)EDTA²⁻ through ion exchange and surface coordination mechanism. However, the adsorption affinity of Pb(II)EDTA²⁻ complex to the surface of HAP was low. The presence of EDTA would reduce the efficiency of the adsorption process, thereby increasing the mobility of Pb²⁺ and potential heavy metal environmental pollution.

It could be seen from Fig. S2 that the presence of HA had a certain effect on the removal of Pb²⁺ by Sr-HAP. When the concentration of HA increased from 0 to 20 mg/L, it would reduce the adsorption of Pb²⁺ on Sr-HAP, which might be due to the saturation of active sites for adsorption on SR-HAP. After the concentration was 20 mg/L, a further increase in the concentration of HA would not lead to the significant change in Pb²⁺ adsorption, and the removal rate remained basically constant. The similar phenomenon had been observed in previous studies (Wang et al. 2008; Zhu et al. 2010). Because of the strong affinity between Pb²⁺ and Sr-HAP, Sr-HAP always acted faster on Pb²⁺ than HA. Even if its concentration increased the driving force for HA to adsorb to the surface of Sr-HAP was still insufficient to compete with Pb²⁺. The effect of FA on the removal of Pb²⁺ was stronger than that of HA, and the overall removal trend of Pb²⁺ was similar to that of HA.

In the concentration range of 0 ~ 200 mg/L, competitive cations (Ca²⁺ and Mg²⁺) had almost no effect on Pb²⁺ immobilized by Sr-HAP (Fig. 9), the removal rate was almost maintained above 99.95%, and the results indicated that SR-HAP was selective to the competitive cations that usually existed in the water environment and could effectively remove the divalent metals. Ca²⁺ was the parent cation of Sr-HAP. Compared with Mg²⁺, Ca²⁺ had the higher affinity for the surface of Sr-HAP. Increasing the initial concentration gradually could be regarded as the increasement in the concentration of adsorbate, but it did not affect the removal rate of Pb²⁺, because the adsorption capacity of SR-HAP for Pb²⁺ had not reached saturation.

**Adsorption–desorption experiment**

The comparison of the desorption of Sr-HAP adsorbent after adsorbing Pb²⁺ with different desorption solutions is shown in Fig. 10. It could be seen that except for TCLP1 (pH 4.93), HCl, EDTA and NaOH could desorb Pb²⁺, the desorption effect of the other desorption agents on the equilibrium Pb²⁺ adsorption was almost zero. The above phenomenon showed that Pb²⁺ could be more stably fixed on Sr-HAP, and the desorption rate of Pb²⁺ with stronger acidic desorption solution (such as HCl) was stronger. In the case of high H⁺ concentration, the chemical reaction of Pb²⁺ on the SR-HAP surface, such as surface complexation, would have an inhibitory effect, resulting in the desorption of Pb²⁺. The other part of the reason was the partial dissolution of apatite group minerals under the condition of pH < 3.0. Due to environmental changes, such as changes in pH, the lead fixed on the surface was easily desorbed into the solution again, while the
precipitated and complexed lead was very stable. It could be seen from Tables 1, 2, 3, 4 that the desorption rate of Pb\(^{2+}\) for most of the desorption liquids after three cycles of adsorption–desorption process was lower than 11.42\%, indicating that Sr-HAP could be used as an adsorbent with excellent performance to immobilize heavy metals in wastewater containing Pb\(^{2+}\).

**Adsorption kinetics**

Among the different kinetic equations, the experimental data of Pb\(^{2+}\) adsorption by Sr-HAP was most suitable for the pseudo-second-order kinetic model, which was described as follows:

\[
\frac{t}{q_t} = \frac{1}{q_e} + \frac{t}{q_e} \quad (2)
\]

In the formula, \(q_t\) and \(q_e\) were the adsorption amount (mg/g) at the time and equilibrium, respectively; \(K_2\) was the pseudo-second-order kinetic rate constant, g/(mg-min).

The adsorption rate (h) (mg/(g min)) could be expressed as:

\[
h = k_2q_e^2 \quad (3)
\]

**Table 1** Elements mass after Sr-HAP adsorption of Pb\(^{2+}\) (A and B are different scanning positions)

| The element | Wt (%) | Atomic percentage | The element | Wt (%) | Atomic percentage |
|-------------|--------|-------------------|-------------|--------|-------------------|
| O           | 23.80  | 69.87             | O           | 26.39  | 69.05             |
| P           | 7.10   | 10.76             | P           | 8.23   | 11.13             |
| Ca          | 2.33   | 2.73              | Ca          | 4.65   | 4.85              |
| Sr          | 4.87   | 2.61              | Sr          | 9.76   | 4.66              |
| Pb          | 61.90  | 14.03             | Pb          | 50.97  | 10.30             |
| The total   | 100%   | 100%              | The total   | 100.00 | 100.00            |

**Table 2** Kinetic fitting parameters of Sr-HAP to Pb\(^{2+}\)

| Simulation type       | T/K   | Rate constant/K | \(q_{e, cal}\) (mg/g) | \(q_{e, exp}\) (mg/g) | \(R^2\)  |
|-----------------------|-------|-----------------|------------------------|------------------------|---------|
| Pseudo-second-order dynamics | 298   | \(K_2\) [g/(mg-min)] = | 4.9898\times10^{-4} | 588.235 | 587.077 | 0.9996 |
|                        | 308   | \(K_2\) [g/(mg-min)] = | 4.9828\times10^{-4} | 588.235 | 593.863 | 0.9998 |
|                        | 318   | \(K_2\) [g/(mg-min)] = | 5.1607\times10^{-4} | 588.235 | 598.138 | 0.9997 |
The equilibrium adsorption capacity ($q_e$) and the pseudo-second-order constant ($K_2$) could be calculated from the slope and intercept of the curve $t/q_t$ vs $t$ (Fig. S3(A)). As shown in Fig. S3(A), the correlation coefficients $R^2$ of the pseudo-second-order kinetic equations were all equal to 1.0, which could better describe the process of removing Pb$^{2+}$ by Sr-HAP. Pseudo-second-order kinetics and Elovich kinetic equations were mainly used to describe chemical adsorption, that is, the process of Pb$^{2+}$ and Sr-HAP binding. The reaction was fast in the chemical adsorption stage, and the rate of Sr-HAP adsorption of Pb$^{2+}$ was mainly controlled by membrane diffusion and internal diffusion.

Table 2 shows the kinetic fitting parameters of Sr-HAP on Pb$^{2+}$. The theoretical equilibrium adsorption capacities obtained by the pseudo-second-order kinetic model were 588.235, 588.235 and 588.235 mg/g, respectively, and the experimental results were 587.077, 593.863 and 598.138 mg/g which were relatively close to the theoretical equilibrium adsorption capacities. Besides, the correlation coefficient $R^2$ was close to 1.

### Adsorption isotherm

For ideal monolayer adsorption, the linear expression of Langmuir isotherm adsorption model was:

$$ \frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{q_m K_L} $$

(4)

$$ R_L = \frac{1}{1 + K_L C_0} $$

(5)

In the formula, $q_e$ was the equilibrium adsorption capacity, mg/g; $q_m$ was the maximum adsorption capacity, mg/g; $C_e$ was the mass concentration of the solution at equilibrium, mg/L; $C_0$ was the initial concentration of the solution, mg/L; $R_L$ was the Langmuir adsorption Equilibrium constant; and $K_L$ was the coefficient of Langmuir adsorption isotherm equation, mg/L.

The correlation coefficients $R^2$ of the Langmuir model were 0.997 (25 °C), 0.998 (35 °C) and 0.999 (45 °C), indicating the uniformity of the adsorbent surface (Table 3 and Fig. S3(B)). The maximum adsorption $q_m$ was 588.235 mg/g (25 °C), 625.000 mg/g (35 °C) and 666.667 mg/g (45 °C), respectively. And the experimental results were 575.248 mg/g (25 °C), 610.037 mg/g (35 °C) and 650.249 mg/g (45 °C), respectively, which were similar to $q_m$. It showed that the Langmuir isotherm adsorption model was very suitable to describe the adsorption behavior of Sr-HAP on Pb(II).

The adsorption equilibrium constant $R_L$ value could indicate the adsorption performance of the material: unfavorable adsorption ($R_L > 1$), favorable adsorption...
(0 < R_L < 1), linear adsorption (R_L = 1), irreversible adsorption (R_L = 0). In Table 4, R_L was between 0 and 1, indicating that the adsorption process of Sr-HAP on Pb^{2+} was favorable.

**Adsorption mechanism**

The downward trend of the final pH change curve in the solution in Fig. 11 was very significant. The rapid removal of Pb^{2+}, the high fixed amount of Pb^{2+} and the specific pH dependence strongly indicated that dissolution/precipitation was the main mechanism of Pb^{2+} removal (Chen et al. 1997). Relevant studies had also showed that the process of removing Pb^{2+} by apatite not only involved surface complex adsorption, ion exchange and dissolution/precipitation, but also depended on the initial lead concentration (Guan et al. 2018). The possible accompanying mechanisms in the process of Sr-HAP removal of Pb^{2+} were: surface complexation, dissolution/precipitation, ion exchange and surface adsorption (Pham et al. 2013). When Pb^{2+} in the solution met Sr-HAP, Pb^{2+} quickly adsorbed to the pores on the surface of the adsorbent, and the functional group (-OH) on the surface of the adsorbent reacted with Pb^{2+} to release H^+; part of the target adsorbed on the adsorbent Pb^{2+} reacted with calcium ions and strontium ions in Sr-HAP. When the adsorbent released H^+, the pH of the solution decreased, Sr-HAP would partially dissolve and release PO_4^{3-}, and Pb^{2+} tended to form Pb_{10}(PO_4)_6(OH)_2 with phosphate. With the progress of the removal process, the available sites on the surface and inside of Sr-HAP were gradually fully occupied, the ionic components of the solution and the surface of the material tended to balance, and the entire removal process was basically completed.

Among them, the surface complexation mechanism could be expressed as:

\[\equiv \text{POH} + \text{Pb}^{2+}_{(aq)} \leftrightarrow \equiv \text{PO Pb}^+ + \text{H}^+_{(aq)}\]  \hspace{1cm} (6)

\[\equiv \text{PO}^- + \text{Pb}^{2+}_{(aq)} \leftrightarrow \equiv \text{PO Pb}^+\]  \hspace{1cm} (7)

\[\equiv \text{CaOH} + \text{Pb}^{2+}_{(aq)} \leftrightarrow \equiv \text{CaOPb}^+ + \text{H}^+_{(aq)}\]  \hspace{1cm} (8)

\[\equiv \text{SrOH} + \text{Pb}^{2+}_{(aq)} \leftrightarrow \equiv \text{SrOPb}^+ + \text{H}^+_{(aq)}\]  \hspace{1cm} (10)

The dissolution/precipitation mechanism was as follows:

**Dissolve:**

\[\text{Sr}_5\text{Ca}_6(\text{PO}_4)_6(\text{OH})_2 + 14\text{H}^+ \rightarrow 5\text{Ca}^{2+} + 5\text{Sr}^{2+} + 6\text{H}_2\text{PO}_4^- + 2\text{H}_2\text{O}\]  \hspace{1cm} (10)

**Precipitate:**

\[10\text{Pb}^{2+} + 6\text{H}_2\text{PO}_4^- + 2\text{H}_2\text{O} \rightarrow 14\text{H}^+ + \text{Pb}_{10}(\text{PO}_4)_6(\text{OH})_2\]  \hspace{1cm} (11)

The ion exchange was:

\[\text{Sr}_5\text{Ca}_6(\text{PO}_4)_6(\text{OH})_2 + (x+y)\text{Pb}^{2+} \rightarrow (5-x)\text{Ca}^{2+} + (5-y)\text{Sr}^{2+} + \text{Sr}_x\text{Ca}_y\text{Pb}_{(x+y)}(\text{PO}_4)_6(\text{OH})_2\]  \hspace{1cm} (12)

**Conclusions**

In this work, a strontium-doped hydroxyapatite (Sr-HAP) was synthesized and used as adsorbent to remove lead ions (Pb^{2+}) from water. The results showed that strontium-doped hydroxyapatite Sr-HAP showed good adsorption capacity for Pb^{2+}. When the dosage of Sr-HAP was 40 mg/50 mL, the removal rate of Pb^{2+} was above 98% at different concentrations (100, 200 and 300 mg/L). And the initial concentration of Pb^{2+} increased from 100 mg/L to 800 mg/L, the removal amount of Pb^{2+} increased from 128.779, 128.481 and 128.474 mg at 25, 35 and 45 °C, respectively. Compared with HAP, the specific surface area of Sr-HAP has increased by 11.1%, from 102.314, 128.779 and 128.481 mg at 25, 35 and 45 °C, respectively. The pore size of Sr-HAP is mostly distributed in 2–50 nm. Meanwhile, the adsorption–desorption experiments show that Pb^{2+} can be relatively stable and fixed in the material, and it is difficult to desorb. Surface complexation, ion exchange, and surface adsorption are the main effects of Pb^{2+} adsorption onto Sr-HAP. In addition, co-precipitation and dissolution/precipitation may exist in the adsorption process of Pb^{2+}.

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Huiling Jiang: Validation, Formal analysis and Visualization.
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Data Availability Not applicable.

Declarations

Ethical Approval Yes. Our study does not involve human subjects.

Consent to Participate Yes.

Consent to Publish Yes.

Competing Interests We declare that we have no financial and personal relationships with other people or organizations that can inappropriately influence our work, and there is no professional or other personal interest of any nature or kind in any product, service and company that could be construed as influencing the position presented in the manuscript.

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