Efficient adsorption of Pb(II) by sodium dodecyl benzene sulfonate intercalated calcium aluminum hydrotalcites: kinetic, isotherm, and mechanisms

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Abstract
Two novel adsorbents of CaAl-LDHs and sodium dodecyl benzene sulfonate (SDBS) intercalated CaAl-LDHs (SDBS-CaAl-LDHs) were successfully prepared by co-precipitation. The main composition and physical properties of two samples were characterized by XRD, XPS, FT-IR, TG, and SEM. Batch adsorption experiments were conducted to study the effect of pH, adsorption time, and initial concentration of Pb²⁺. The results showed that the prime adsorption conditions obtained were pH of 5.2 after 60 min with the initial concentration of 300 mg g⁻¹ for CaAl-LDHs and 350 mg g⁻¹ for SDBS-CaAl-LDHs. At 303 K, the adsorption capacities and removal rates of CaAl-LDHs and SDBS-CaAl-LDHs were found to be 456.05 mg g⁻¹, 91.21% and 682.26 mg g⁻¹, 97.47%, respectively. For CaAl-LDHs, the kinetic data for Pb²⁺ was best fitted with pseudo-2nd-order model, and the adsorption isotherms followed Langmuir and Freundlich isotherm model. The adsorption data of SDBS-CaAl-LDHs can be best described by the pseudo-second-order kinetic and Langmuir model. The Pb²⁺ adsorption mechanism on SDBS-CaAl-LDHs was explored by XRD, XPS, and SEM, and the important roles of the electrostatic attraction, precipitation, complexation, and ion exchange were demonstrated. The Langmuir adsorption capacities for SDBS-CaAl-LDHs were 797.63, 828.76, and 854.29 mg g⁻¹ at 293 k, 303 k, and 313 k, respectively. Thus, SDBS-CaAl-LDHs may be a highly economical adsorbent for the treatment of contaminated water.

Keywords Adsorption mechanism · Adsorption isotherm · CaAl-LDHs · Pb²⁺ · SDBS-CaAl-LDHs

Introduction
Water pollution is a global problem because of the discharge of various organic and inorganic contaminants such as dyes (Rashid et al. 2021a, b; Azam et al. 2020; Shabir et al. 2022), sulfur-containing compounds (Rashid et al. 2021a, b), heavy metals (Rashid et al. 2021a, b), surfactants, pesticides, and pharmaceuticals (Basheer 2018; Basheer and Ali 2018; Basheer and Arsh 2018; Ali and Jain 1998; Ali et al. 2011). In China, 68 pollutants are listed as priority pollutants, which include 58 toxic organic and some inorganic pollutants, such as heavy metals and their compounds. Pb²⁺, Hg²⁺, and Cd²⁺ are the most common heavy metal pollution in water.

Pb²⁺ in the aquatic environment is a serious threat to human health due to their bioaccumulation, toxicity, and potential biomagnifications (Kumar et al. 2017). The maximum concentration of Pb²⁺ in central drinking water in the environmental quality standards for surface water (GB 3838–2002) is ≤0.05 mg L⁻¹, while the maximum discharge concentration of Pb²⁺ for industrial effluents in the integrated wastewater discharge standard (GB8978-1996) is 1.0 mg L⁻¹ in China, respectively. Therefore, the removal of Pb²⁺ from wastewater is important to mitigate the environmental and human health impacts of heavy metal.
The common technologies for pollutant removal are adsorption (Tian et al. 2020), chemical precipitation (Chen et al. 2018), electrolysis (Tao et al. 2014), ion exchange, membrane separation (Berbar et al. 2019), phytoremediation (Wang et al. 2018), and biological flocculation. The adsorption method is considered a suitable technique for wastewater treatment because it has a simple design, good adsorption efficiency, and low cost (Ali et al. 2005; Ali and Aboul-Enein 2002; Ali et al. 2021a, b, c). Design and fabrication of advanced materials for effective and efficient adsorption is an attractive research field (Basheer 2020; Ali et al. 2018, 2019a, b). Some advanced materials, such as modified montmorillonite and activated carbon, have been found to remove organic pollutants (Ali et al. 2020, 2021a, b, c). Ali et al. (2019a, b) reported removal of Cu(II) and Zn(II) metal ions in water by advanced material of polyhydroquinone/graphene nanocomposite. The result showed that the uptake time for Cu(II) and Zn(II) at pH 6.0 was 60 and 15 min, respectively, indicating fast hydro-friendly nature and making the method applicable to solve water pollution.

Adsorption of heavy metal ions (such as lead, cerium and scandium) using cost-efficient adsorbents has been studied in the last few years, such as bentonite (Niu et al. 2020; Burakova et al. 2018), activated carbon (Liu et al. 2019), fly ash (Huang et al. 2020), clay (Abukhadora et al. 2019), and zeolite (Han et al. 2020). These adsorbents have slow adsorption kinetics and low adsorption capacities for adsorbing heavy metal ions from wastewater (Gupta et al. 2010; Ali et al. 2021a, b, c). Therefore, it is necessary to employ novel materials that have significantly higher adsorption capability, stable structure, and additionally modified with various chemically active groups to enhance adsorption activity and selectivity.

Layered double hydroxides (LDHs) are considered to be a class of anion clay with layered structure. Their general formula are \([\text{M}^{2+}\text{M}^{3+}\text{X}^{-}\text{H}_{2}\text{O}]\), where \(\text{M}^{2+}\) and \(\text{M}^{3+}\) represent divalent and trivalent metal ion, and \(\text{X}^{-}\) is an interlayer anion (Grover et al. 2019). Because of its ion-exchange ability, adjustable element proportions, special-layered structure, and operation of simple synthesis, LDHs and modified materials of LDHs were widely studied in wastewater treatment. For example, Yang et al. (2016) used Mg–Al layered double hydroxide-modified palygorskite (Pal/MgAl-LDH) as adsorbent to remove Pb\(^{2+}\), Cu\(^{2+}\), and Ni\(^{2+}\) with the maximum adsorption capacity of 99.8, 64.0, and 41.8 mg g\(^{-1}\) at pH 5.0, respectively. Jia et al. (2019) utilized magnetic biochar supporting MgFe-LDH composites to remove Pb\(^{2+}\) and found that the maximum adsorption capacity is 476.25 mg g\(^{-1}\); the adsorption process mainly contains chemisorptions. Zhu et al. (2020a, b) studied the removal of Cu\(^{2+}\) and Pb\(^{2+}\) by amino trimethylene phosphonic acid (ATMP) intercalated Zn–Al-layered double hydroxide, and the adsorption capacity for Cu\(^{2+}\) and Pb\(^{2+}\) were 42.02 and 84.06 mg g\(^{-1}\).

The homogeneous dispersion of LDHs is a technical challenge because the layered surface of LDHs contains a large number of active hydroxyl groups. To improve the dispersion of LDHs, a possible solution is transforming the hydrophilic LDHs into a hydrophobic one by surfactant-intercalated LDHs such as sodium dodecyl sulfate (SDS) (Ahmed et al. 2015; Zhang et al. 2012), sodium dodecyl benzene sulfate (SDBS) (Zhang et al. 2014), and sodium hexadecyl sulfate (SHS) (Zhang et al. 2015) into LDH interlayer spaces. In the literature, the synthesis of surfactant-intercalated LDHs had been extensively used to remove organic contaminants such as pesticides (Bruna et al. 2006), herbicides (Carrizosa et al. 2004), dyes (Zhang et al. 2017; Zhu et al. 2020a, b), and phenols (Zaghouane-boudiaff et al. 2011). However, to the best of our knowledge, the removal of heavy metal ions from aqueous solutions was not reported elsewhere by surfactant-intercalated Ca–Al-layered double hydroxides (CaAl-LDHs). The detailed effect of the structure of interlayer anions on metal ion adsorption by CaAl-LDHs has not been reported. It is expected that the combination of suitable inter-layer anion with the LDH layers can produce materials capable of possessing suitable binding sites to remove heavy metal ions.

Therefore, the present work has proposed to use CaAl-LDHs and SDBS intercalated CaAl-LDHs as an adsorption material for lead removal. The structure and morphology of CaAl-LDHs and SDBS-CaAl-LDHs were characterized by Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), and scanning electron microscopy (SEM). In the first step, adsorption isotherm studies were performed. Then, the sample solution pH and adsorption time affecting the performance of the target metal ion adsorption were evaluated, as well as the extraction efficiency of CaAl-LDHs and SDBS-CaAl-LDHs. Furthermore, the XRD SEM and XPS techniques were investigated to evaluate the interaction mechanism of lead ions with the SDBS-CaAl-LDHs.

**Experimental section**

**CaAl-LDH preparation**

The CaAl-LDHs with a Ca/Al ratio of 3:1 were synthesized by co-precipitation, according to the method described by Wang and Grover (Grover et al. 2019; Wang et al. 2020). In brief, first, CaO, Al(NO\(_3\))\(_3\)-9H\(_2\)O, and urea were separately dispersed in deionized water with ultrasonication for 0.5 h; next, Al(NO\(_3\))\(_3\) and urea solution was added to the suspension of Ca(OH)\(_2\) drop by drop while refluxing at 100 °C on a magnetic
stirrer. After that, this slurry was filtered, washed, dried, and ground into powder (mesh size is 30), namely, CaAl-LDHs.

**SDBS-CaAl-LDH preparation**

CaAl-LDH power was calcined at 400 °C for 2 h to obtain layered double oxides (CaAl-LDO). A total of 2.8 g CaAl-LDO was added to 5.8 g SDBS solution at room temperature under magnetic stirring and transferred into autoclave at 150 °C for 1.5 h. Afterward, the sample was filtrated and washed at normal temperature and dried at 60 °C in an oven. The finished product of SDBS-CaAl-LDHs is finally obtained by sieving.

**Batch sorption experiments**

To estimate the adsorption capacity and removal rate of adsorbent, constant amount of SDBS-CaAl-LDHs and CaAl-LDHs was added to Pb²⁺ aqueous solutions. The mixture was shaken by the thermostated shaker for a sufficient time at a specified temperature. To reach adsorption equilibrium, the suspensions were easily separated, and the lead ion concentration residue was tested. Adsorption capacity and removal rate of lead ions can be estimated using Eqs. (1) ~ (3):

\[
 Q_e = \frac{(C_0 - C_e)}{m} \text{V} \quad (1)
\]

\[
 Q_t = \frac{(C_0 - C_t)}{m} \text{V} \quad (2)
\]

\[
 R\% = 100\% \times \frac{(C_0 - C_e)}{C_0} \quad (3)
\]

where \( Q_e \) and \( Q_t \) (mg g⁻¹) are the adsorption capacity of lead ions adsorbed at equilibrium time and time \( t \); \( C_0 \), \( C_e \), and \( C_t \) (mg L⁻¹) are the lead ion concentration at the initial time, equilibrium time, and time \( t \), respectively; \( m \) (g) is the weight of adsorbent; \( V \) (L) is the volume of lead ion solution; \( R\% \) is removal rate.

**Characterization**

The characteristics of SDBS-CaAl-LDHs and CaAl-LDHs and the mechanisms for adsorbing Pb²⁺ were inspected by SEM (Sigma HD), XRD (X’ Pert PRO), FT-IR (Shimadzu Corporation, Japan), and XPS (Thermo Scientific K-Alpha +). The thermal stability of CaAl-LDHs and SDBS-CaAl-LDHs was characterized using 209F3 thermal analyzer.

**Error analysis**

Non-linear regression analysis for adsorption isotherms was performed. Standard deviation (SD) functions (see the formula 4) (Hami et al. 2020) studied the estimation of the best-fitting isotherm as shown in Table 1:

\[
 SD = \sqrt{\frac{1}{n} \sum_{i=1}^{n} (Q_{cal} - Q_{exp})^2} \quad (4)
\]

**Results and discussion**

**General characterization**

The FT-IR spectrum of CaAl-LDHs and SDBS-CaAl-LDHs is investigated in Fig. 1. A peak at 3400–3700 cm⁻¹ and a weak peak at 1625 cm⁻¹ were related to the –OH stretching and bending vibrations of the metal hydroxide layers and interlayer water molecules, and a peak at 1381 cm⁻¹ was ascribed to the \( \text{NO}_3^- \) ion stretching vibration (Gupta et al. 2020). The very strong peak at 1355 cm⁻¹ and 1407 cm⁻¹ represents \( \text{CO}_3^{2-} \) stretching mode (Jia et al. 2019). Several peaks observed in 1000–400 cm⁻¹ mainly belonged to characteristic peaks of Ca–O and Al–O bonds (Sun et al. 2017). Two bands located at 2982 and 2873 cm⁻¹ were due to the C–H stretching vibration bands of dodecyl sulfate ions in SDBS-CaAl-LDHs (b). The sulfate S=O stretch at 1187 and 1066 cm⁻¹ was also observed (Bouraada et al. 2008).

The decomposition temperatures and mass loss of CaAl-LDHs and SDBS-CaAl-LDHs have been performed by the TG-DTG analysis (Fig. 2). The TG-DTG curve of CaAl-LDHs (Fig. 2a) showed the major mass loss stages. The initial weight loss of 8.21% before 165 °C (step 1) was due to the loss of surface adsorbed water and step 2 at 165–425 °C, bigger than step 1 (weight loss of 16.24% for step 2), to the loss of interlayer water; weight loss of 17.45%

| Equilibrium models | Parameters | Adsorbent | 293 K | 303 K | 313 K |
|--------------------|------------|-----------|-------|-------|-------|
| **Langmuir**       |            |           |       |       |       |
| \( Q_m \) (mg g⁻¹) | i          | 717.17    | 815.07| 782.51|       |
|                    | ii         | 797.63    | 828.76| 854.29|       |
| \( K_L \) (L g⁻¹)  | i          | 0.0547    | 0.0507| 0.0644|       |
|                    | ii         | 0.2904    | 0.4968| 0.5694|       |
| \( S_D \)          | i          | 0.9689    | 0.9746| 0.9764|       |
|                    | ii         | 0.9852    | 0.9845| 0.9899|       |
| **Freundlich**     |            |           |       |       |       |
| \( K_f \) (mg g⁻¹) | i          | 101.16    | 95.95 | 109.27|       |
|                    | ii         | 424.99    | 473.35| 495.72|       |
| \( n \)            | i          | 0.4131    | 0.4637| 0.4394|       |
|                    | ii         | 0.1373    | 0.1329| 0.1314|       |
| \( R^2 \)          | i          | 0.9723    | 0.9827| 0.9776|       |
|                    | ii         | 0.8970    | 0.9195| 0.8740|       |
in step 3 above 425 °C is lattice water, the decomposition of \( \text{CO}_3^{2-} \) and dehydroxylation from CaAl-LDH layers. The total weight loss for CaAl-LDHs was 41.90%. Compared with the curve of CaAl-LDHs (Fig. 2a), the TG-DTG curve of SDBS-CaAl-LDHs (Fig. 2b) showed two major mass loss stages. The initial weight loss of 11.80% before 540 °C was due to the loss of adsorbed water and lattice water and the decomposition of the intercalated SDBS and \( \text{CO}_3^{2-} \) from LDHs of SDBS-CaAl-LDHs (Sun et al. 2017; Mahjoubi et al. 2019). The weight loss of 29.22% from 540 to 800 °C in the second step corresponded to the dehydroxylation of SDBS-CaAl-LDH layers (Sun et al. 2017). The total weight loss for SDBS-CaAl-LDHs was 40.02%. Therefore, the residue amount in the CaAl-LDHs was much lower than that of the SDBS-CaAl-LDH material.

Adsorption isotherms

Adsorption isotherms were useful to provide qualitative information on the equilibrium adsorption capacity of CaAl-LDHs and SDBS-CaAl-LDHs and equilibrium concentration of lead ions between the solid and liquid phase. Herein, equilibrium adsorption isotherms, such as Langmuir and Freundlich, were applied to represent the adsorption data of lead ions on CaAl-LDHs and SDBS-CaAl-LDHs powders. They were given in Eqs. (5 and 6) (Langmuir 1918; Freundlich 1907):

\[
Q_e = \frac{K_L Q_m C_e}{(1 + K_L C_e)} \tag{5}
\]

\[
Q_e = K_f C_e^n \tag{6}
\]

where \( Q_m \) (mg g\(^{-1}\)) and \( K_f \) (L mg\(^{-1}\)) denote the maximum adsorption capacity related to the complete monolayer coverage of CaAl-LDHs and SDBS-CaAl-LDHs and the Langmuir adsorption equilibrium constant corresponding to the adsorption energy. \( K_f \) (mg\(^{1-n}\) L\(^n\) g\(^{-1}\)) and \( n \) (dimensionless constant) are Freundlich constant corresponding to the adsorption capacity and adsorption intensity, respectively. If the values of \( n \) are between 0 and 1, it indicates a favorable adsorption (Zeng et al. 2018, 2019).

The removal rate and Freundlich and Langmuir isotherms for different initial \( \text{Pb}^{2+} \) concentration adsorption on the synthesized CaAl-LDHs and SDBS-CaAl-LDHs are shown in Fig. 3a–h. As shown in Fig. 3a–d, the removal rate (R%) of CaAl-LDHs and SDBS-CaAl-LDHs was decreased gradually with the increase of \( \text{Pb}^{2+} \) concentration; however, the sorption capacity \( (Q_e) \) was exactly opposite at three temperatures. The \( \text{Pb}^{2+} \) removal rate (R%) by CaAl-LDHs was lower than that by SDBS-CaAl-LDHs at the same dosage. For example, when the dosage was 0.5 g L\(^{-1}\), the \( \text{Pb}^{2+} \) removal rate of CaAl-LDHs and SDBS-CaAl-LDHs was 87.11% and 98.84% as 300 mg L\(^{-1}\) for \( \text{Pb}^{2+} \) initial concentration at 303 K. In addition, the \( Q_e \) of CaAl-LDHs and SDBS-CaAl-LDHs increased with the initial concentration of \( \text{Pb}^{2+} \), and \( Q_e \) increased.

![Fig. 1 FT-IR spectra of CaAl-LDHs (a) and SDBS-CaAl-LDHs (b)](image1)

![Fig. 2 TG and DTG curves of CaAl-LDHs (a) and SDBS-CaAl-LDHs (b)](image2)
more swiftly at low concentration of Pb\(^{2+}\). This showed that a lot of lead ions were deposited on the surface of adsorbent with the increase of Pb\(^{2+}\) concentration. From the experimental data, the \(Q_e\) of SDBS-CaAl-LDHs in 303 K is 682.26 mg g\(^{-1}\) (\(C_0 = 350\) mg L\(^{-1}\), \(m = 0.05\) g) which is 1.496 times higher than the \(Q_e\) (456.05 mg g\(^{-1}\)) of CaAl-LDHs (\(C_0 = 300\) mg L\(^{-1}\), \(m = 0.06\) g). It also showed that surfactant-intercalated CaAl-LDHs were a significant modification method to improve the adsorption capacity.

Figure 3e – h are Freundlich and Langmuir isotherms. Table 1 presents the constants (\(Q_m, K_L, K_f, n\)) and correlation coefficients (\(R^2\)) of Langmuir and Freundlich isotherm models of CaAl-LDHs and SDBS-CaAl-LDHs for the adsorption of Pb\(^{2+}\). The results demonstrated that the adsorption data of
Pb$^{2+}$ on CaAl-LDHs were fitting well with the Langmuir and Freundlich model; the correlation coefficient $R^2$ of Langmuir and Freundlich model was 0.9689, 0.9746, and 0.9764 and 0.9723, 0.9827, and 0.9776 at three temperatures, respectively. The experimental data of Pb$^{2+}$ on SDBS-CaAl-LDHs indicated the Langmuir model had higher fitting than the Freundlich model; the correlation coefficient $R^2$ of Langmuir model was 0.9852, 0.9845, and 0.9899 at three temperatures, respectively. The Langmuir isotherm SD of CaAl-LDHs was higher than that of SDBS-CaAl-LDHs. Thus, Langmuir model for SDBS-CaAl-LDHs was the suitable isotherm.

The Langmuir adsorption capacities ($Q_m$) for CaAl-LDHs and SDBS-CaAl-LDHs were 717.17, 815.07, and 782.51 and 797.63, 828.76, and 854.29 mg g$^{-1}$, respectively. The $K_L$ value of SDBS-CaAl-LDHs was significantly higher than that of CaAl-LDHs, suggesting that SDBS-CaAl-LDHs had strong adsorption ability (Jia et al. 2019). The value of $n$ for two materials was below 1; the lower value of $n$ of SDBS-CaAl-LDHs than of CaAl-LDHs suggested that SDBS-CaAl-LDHs had better adsorption performance.

$Q_m$ of Pb$^2+$ for SDBS-CaAl-LDHs and CaAl-LDHs was compared with the other adsorbents in previous studies (Hou et al. 2020; Dinari and Neamati 2020; Zhao et al. 2011), and the results are listed in Table 2. It was clear that SDBS-CaAl-LDHs had a better adsorption capacity than other materials.

### Adsorption kinetics

The contact time, as one of the important factors for the analysis of lead removal on adsorbent (SDBS-CaAl-LDH, CaAl-LDH), was studied (Fig. 4). The results showed that removal rate ($R%$) (Fig. 4a, b) and adsorption capacity ($Q_c$) on the adsorbent for Pb$^{2+}$ were rapidly growing within 30 min, then slowly increased from 30 to 60 min and remained unchanged later. The results could be proposed for the removal mechanism: due to the many adsorption sites at the initial stage, which could be available for the fast Pb$^{2+}$ sorption from the solution to the external surfaces of adsorbents (SDBS-CaAl-LDH, CaAl-LDH), this sorption process was considered for the rapid diffusion of Pb$^{2+}$; then, slow sorption was owed to slow diffusion of Pb$^{2+}$ from the boundary layer to the interlayer space of adsorbent and replacing Ca(II) by Pb$^{2+}$ on the interlayer surface of SDBS-CaAl-LDH or CaAl-LDH as reported (Chen et al. 2016). Therefore, sorption curves increased slowly by slow diffusion at a later stage. As time proceeds, Pb$^{2+}$ diffused into the porous structure of SDBS-CaAl-LDH or CaAl-LDH leading to little variation for sorption curves.

Rapid adsorption was a very useful criterion for identifying the adsorption efficiency of the adsorbent. Generally, two different models (the pseudo-first-order and pseudo-second-order models) were served to investigate the kinetic behavior of Pb$^{2+}$ on CaAl-LDHs and SDBS-CaAl-LDHs; two kinetics equation could be shown (Zeng et al. 2018, 2019):

$$Q_t = Q_e(1 - \exp(K_1t))$$

$$Q_t = (tK_2Q_e^2)/(1 + tK_2Q_e)$$

where $K_1$ (min$^{-1}$) and $K_2$ (g mg$^{-1}$ min$^{-1}$) represent rate constants for pseudo-1st-order and pseudo-2nd-order models, respectively.

In order to complete lead ion removal by two adsorbents, adsorption kinetic curves in Fig. 4c-f were fitted, and the kinetic model corresponding parameters are obtained in Table 3. Calculated sorption capacity ($Q_{e,cal}$) for CaAl-LDHs and SDBS-CaAl-LDHs in pseudo-2nd-order models was 460.41, 466.76, and 471.47 and 699.43, 710.22, and 714.19 mg g$^{-1}$ at 293, 303, and 313 K, respectively. $Q_{e,cal}$ for CaAl-LDHs and SDBS-CaAl-LDHs in pseudo-1st-order models were 446.91, 454.06, and 458.78 and 660.00, 674.36, and 679.77 mg g$^{-1}$ at 293, 303, and 313 K, respectively. Experimental sorption capacity ($Q_{e,exp}$) for CaAl-LDHs and SDBS-CaAl-LDHs was 457.32, 464.85, and 470.70 and 681.26, 685.70, and 692.90 mg g$^{-1}$ at 293, 303, and

### Table 2 Comparison of maximum adsorption capacity ($Q_m$) of Pb$^{2+}$ with other adsorbents

| Adsorbent                  | $Q_m$ (mg g$^{-1}$) | Isotherm   | pH   | Reference                  |
|----------------------------|---------------------|------------|------|----------------------------|
| Mag-LDO/C                  | 359.7               | Langmuir   | 6    | Hou et al. (2020)          |
| ATMP/Zn-Al LDH             | 164.84              | Langmuir   | 5.0  | Zhu et al. (2020a, b)      |
| magnetic bioclay/MgFe-LDH  | 476.25              | Langmuir   | 3–6  | Jia et al. (2019)          |
| Mg2Al-LDH                  | 66.16               | Langmuir   | 7.0  | Zhao et al. (2011)         |
| Pal/MgAl-LDH               | 281.0               | Langmuir   | 5    | Yang et al. (2016)         |
| PANI/LDH NCs               | 109.71              | Langmuir and Freundlich | 6.0  | Dinari and Neamati (2020)  |
| CaAl-LDHs                  | 815.07              | Langmuir and Freundlich | 5.2  | This Study                 |
| SDBS-CaAl-LDHs             | 854.29              | Langmuir   | 5.2  | This Study                 |

**Mag-LDO/C** magnetic layered double oxide/carbon, **ATMP/Zn-Al LDH** amino trimethylene phosphonic acid intercalated layered double hydroxide, **LS** sulfonated lignin, **LDH** layered double hydroxides, **PAL/MgAl-LDH** Mg–Al layered double hydroxide-modified palygorskite, **PANI/LDH NCs** surface-modified Ca/F-layered double hydroxide (LDH)/polyaniline nanocomposites
Fig. 4 Effect of contact time on the adsorption of Pb^{2+} by CaAl-LDHs (a) and SDBS-CaAl-LDHs (b) and their adsorption kinetics (c~f) (conditions: \(C_0 = 300\) mg L\(^{-1}\), \(m = 0.06\) g for CaAl-LDHs; \(C_0 = 350\) mg L\(^{-1}\), \(m = 0.05\) g for SDBS-CaAl-LDHs)

Table 3 Kinetic model parameters for adsorption of Pb^{2+} onto CaAl-LDHs (i) and SDBS-CaAl-LDHs (ii) at different temperature

| Kinetic models        | Parameters         | Adsorbent | 293 K | 303 K | 313 K |
|-----------------------|--------------------|-----------|-------|-------|-------|
|                       | \(Q_{e,exp} (mg \cdot g^{-1})\) | i         | 457.32| 464.85| 470.73|
|                       |                    | ii        | 681.26| 693.10| 693.90|
| Pseudo-first order    | \(Q_{e,cal} (mg \cdot g^{-1})\) | i         | 446.91| 454.06| 458.78|
|                       |                    | ii        | 660.00| 674.36| 679.77|
|                       | \(K_1 (min^{-1})\) | i         | 0.2979| 0.3150| 0.3244|
|                       |                    | ii        | 0.1800| 0.1941| 0.2024|
|                       | \(R^2\)            | i         | 0.8605| 0.8779| 0.8421|
|                       |                    | ii        | 0.8812| 0.9539| 0.9514|
| Pseudo-second-order   | \(Q_{e,cal} (mg \cdot g^{-1})\) | i         | 460.41| 466.76| 471.47|
|                       |                    | ii        | 699.43| 710.22| 714.19|
|                       | \(K_2 (\times 10^{-3} \, g \cdot mg^{-1} \cdot min^{-1})\) | i         | 1.5085| 1.6430| 1.6882|
|                       |                    | ii        | 0.4411| 0.4933| 0.5236|
|                       | \(R^2\)            | i         | 0.9987| 0.9719| 0.9702|
|                       |                    | ii        | 0.9848| 0.9801| 0.9809|
Sorption capacity was contrasted with the value of $Q_{e,\text{cal}}$ by pseudo-1st-order and pseudo-2nd-order models, and $Q_{e,\text{exp}}$, $Q_{e,\text{cal}}$ for pseudo-2nd-order models were close to $Q_{e,\text{exp}}$. In addition, the correlation coefficient $R^2$ ($R^2 = 0.970 \sim 0.999$) of two adsorbents for pseudo-2nd-order models was more than that of pseudo-1st-order model ($R^2 = 0.842 \sim 0.954$), respectively. The results showed that the pseudo-2nd-order model was more suitable than the pseudo-1st-order model to describe the sorption of lead ions on CaAl-LDHs and SDBS-CaAl-LDHs, suggesting that the chemisorption was a rate-limited process (Zhang et al. 2017).

**Effect of pH on sorption of Pb$^{2+}$**

Zeng et al. (2019) showed that lead may form precipitation of Pb(OH)$_2$ at pH greater than 8. Thus, the pH values in this study were studied from 2.0 to 7.0, respectively, and the results are listed in Fig. 5. The adsorption capacity ($Q_e$) and removal rate ($R\%$) of CaAl-LDHs and SDBS-CaAl-LDHs for Pb$^{2+}$ increased significantly as the pH increased from 2.0 to 5.0, the $Q_e$ and $R\%$ of two adsorbents increased slowly as the pH increased from 5.0 to 7.0, and the $Q_e$ and $R\%$ of SDBS-CaAl-LDHs were higher than of CaAl-LDHs. Lead ion sorption came to equilibrium at pH of about 5.2 (the solution pH); the adsorption capacities of CaAl-LDHs and SDBS-CaAl-LDHs were achieved 456 mg g$^{-1}$ (the dosage of 0.6 g L$^{-1}$) and 682 mg g$^{-1}$ (the dosage of 0.5 g L$^{-1}$) at 303 K, respectively. Therefore, SDBS-CaAl-LDHs exhibited the highest activity of lead ion adsorption in the solution. $Q_e$ and $R\%$ were low at low pH; it may be due to the fact that hydrogen ions took up the available binding sites of CaAl-LDHs and SDBS-CaAl-LDHs. The similar result was obtained in the literature (Hou et al. 2020).

**Effect of adsorbent dosage**

Figure 6 shows the effect of the adsorbent dosage for adsorption of Pb$^{2+}$ on CaAl-LDHs (the initial lead ion concentration of 300 mg L$^{-1}$) and SDBS-CaAl-LDHs (the initial lead ion concentration of 350 mg L$^{-1}$). Figure 6a shows that the removal ratio ($R\%$) increased rapidly from 75.7 to 91.2% with the increase of CaAl-LDH dosage (0.20 ~ 0.6 g L$^{-1}$) and then increased slowly from 91.2 to 95.7% with the increase of CaAl-LDH dosage (0.6 ~ 1.0 g L$^{-1}$). Figure 6b shows that the removal ratio ($R\%$) increased rapidly from 63.5 to 97.5% with the increase of SDBS-CaAl-LDH dosage (0.20 ~ 0.5 g L$^{-1}$) and then increased slowly from 97.5 to 98.8% with the increase of SDBS-CaAl-LDH dosage (0.5 ~ 0.7 g L$^{-1}$). The initial increase in removal ratio was due to the additional adsorption sites of CaAl-LDHs and SDBS-CaAl-LDHs. However, the removal ratio was slightly changed when a high dosage of CaAl-LDHs and SDBS-CaAl-LDHs was used, due to the limited availability of lead ion in the medium. In Fig. 6, it could be found that the lead ion adsorption capacity ($Q_e$) of CaAl-LDHs and SDBS-CaAl-LDHs was gradually reduced with the increase of adsorbent dosage. Therefore, the optimum sorbent dosage of CaAl-LDHs and SDBS-CaAl-LDHs was set at 0.6 and 0.5 g L$^{-1}$ in view of efficiency and economy, respectively.

**Adsorption mechanisms**

Lead ion was absorbed on CaAl-LDHs and SDBS-CaAl-LDHs by three paths: (i) external surface adsorption of lead ions by electrostatic interactions and complexation, (ii) replacement of interlayer calcium ions by cation exchange, (iii) formation of Pb precipitates.

XRD patterns of CaAl-LDHs and SDBS-CaAl-LDHs before and after lead ion adsorption are shown in Fig. 7. The diffraction peak positions of CaAl-LDHs and SDBS-CaAl-LDHs could be compared with the pure hexagonal phase.
recorded on PDF 78–1219 (Zhang et al. 2012). The diffraction peak positions of CaAl-LDHs (Fig. 7a) and SDBS-CaAl-LDHs (Fig. 7c) have a slight deviation; peaks were discovered at about 10° (002), 21° (004), 30° (006), and 56° (110). Sharp peaks at around 30° might also be attributed to calcium carbonate (JCPDS 17–863). Peak intensity for SDBS-CaAl-LDHs in (002) and (004) was higher than that of CaAl-LDHs. After Pb2+ ions adsorption (Fig. 7b, d), the XRD diffraction peak had found the new phase of lead hydroxide nitrate (Pb3(OH)5NO3, JCPDS 22–0659) (Rojas 2014) and Pb3(CO3)2(OH)2 (JCPDS 13–013) (Jia et al. 2019) from surface co-precipitation between Pb2+ and interlayer CO3^2−, surface hydroxyl groups of CaAl-LDHs and SDBS-CaAl-LDHs. This might be related to dissolution–precipitation mechanism of Ca–Al layered double hydroxides in the solution (Zhang et al. 2012; Rojas 2014).

The SEM images of CaAl-LDHs and SDBS-CaAl-LDHs before and after lead ions adsorption are shown in Fig. 8. CaAl-LDHs (Fig. 8a) were formed from the approximately hexagonal platy crystallites. Compared with CaAl-LDHs (Fig. 8a), the resulting solid of SDBS-CaAl-LDHs (Fig. 8c) discovered obvious changes in image, in which agglomerated plate-shaped particles were observed. SDBS-CaAl-LDHs exhibited slightly irregular shapes because of some aggregations. The result showed that the morphology of LDHs can be influenced in presence of large organic anions. The SEM after adsorption (Fig. 8b, d) showed that CaAl-LDH and SDBS-CaAl-LDH surface adsorbed lead ions by forming Pb precipitates. The layered structure of particle plates for CaAl-LDHs and SDBS-CaAl-LDHs changed, which was just identical with the XRD results.

The elemental compositions of CaAl-LDHs and SDBS-CaAl-LDHs before and after lead ions adsorption were studied by XPS spectrum (Fig. 9). As shown in Fig. 9a, CaAl-LDHs were not only the predominant elements of O, N, Ca, and Al but also C element from pore expanding agent of urea. Compared with CaAl-LDHs, SDBS-CaAl-LDHs (Fig. 9a) discovered S element, which demonstrated that the SDBS was intercalated into CaAl-LDHs. To identify the changed chemical environment of CaAl-LDHs and SDBS-CaAl-LDHs before and after lead ion adsorption, some complementary XPS experiments (Al, Ca, O, Pb elements) were checked as shown in Fig. 9b~d. As can be seen from Fig. 9b, the binding energy of Ca 2p has two peaks at around 346 and 350 eV (346.98 and 350.38 eV for CaAl-LDHs, 346.98 and 350.48 eV for CaAl-LDHs-Pb, 346.78 and 350.28 eV for SDBS-CaAl-LDHs, 346.68 and 350.18 eV for SDBS-CaAl-LDHs-Pb). Compared with the binding energy of Ca 2p for CaAl-LDHs and SDBS-CaAl-LDHs before and after lead ion adsorption, the results showed that two peaks of Ca 2p from SDBS-CaAl-LDHs had changed whereas a peak from CaAl-LDHs had changed. The binding energies of Al 2p for CaAl-LDHs and SDBS-CaAl-LDHs (Fig. 9c) were 73.98 and 73.88 eV, respectively, decreasing with 0.1 eV for SDBS-CaAl-LDHs. However,
compared with the binding energy of Al 2p after sorption Pb(II) ion, shifts in binding energy (reduction of 0.1 eV for CaAl-LDHs and increase of 0.1 eV for SDBS-CaAl-LDHs) were observed. Two XPS peaks for CaAl-LDHs (138.28 and 143.18 eV) and SDBS-CaAl-LDHs (138.58 and 143.58 eV) after sorption Pb(II) ion (Fig. 9a, d) were observed which

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resulted from Pb 4f absorption. This result showed the existence of both Pb species in the precipitates, such as formation of hydroxides and carbonates for lead ions. The XPS result was in agreement with the XRD result (Fig. 7). This result also showed that the main reactions in the lead ion removal by SDBS-CaAl-LDHs and CaAl-LDHs were precipitation and surface adsorption and were consistent with the literature report (Park et al. 2007). These slight shift in binding energies proves the dominance of complexion mechanism (Gupta et al. 2020) Thus, SDBS-CaAl-LDHs or CaAl-LDHs formed complex with lead ions through specific adsorption (such as surface complexion) and non-specific adsorption (such as electrostatic interactions including cation exchange) as described in Eqs. (9 and 10) (Li et al. 2017) and Fig. 10.

\[
\text{Sur} - \text{OH} + \text{Pb}^{2+} \rightarrow \text{Sur} - \text{O} - \text{Pb}^+ + \text{H}^+ \text{specific adsorption (9)}
\]

\[
\text{Sur} - \text{OH} + \text{Pb}^{2+} \rightarrow \text{Sur} - \text{O}... \text{Pb}^{2+} + \text{H}^+ \text{non-specific adsorption (10)}
\]

**Conclusions**

In this study, we showed that SDBS-CaAl-LDHs showed much higher adsorption ability for refining Pb\(^{2+}\) than that of CaAl-LDHs. The precipitates of hydroxides and carbonates for Pb\(^{2+}\) confirmed the restacking of the LDH layers by XRD pattern after wastewater treatment. The adsorption of lead ions by CaAl-LDHs and SDBS-CaAl-LDHs was in keeping with the pseudo-second-order kinetic and Langmuir model. The Langmuir adsorption capacities for CaAl-LDHs and SDBS-CaAl-LDHs were 717.17, 815.07, and 782.51 mg g\(^{-1}\) and 797.63, 828.76, and 854.29 mg g\(^{-1}\), respectively. XPS and XRD results showed that the adsorption occurred via precipitation, electrostatic interactions, isomorphic substitution, and surface complexion. These findings are important for developing LDH-type adsorbents for heavy metal wastewater purification. Collectively, SDBS-CaAl-LDHs may be a highly economical adsorbent for the treatment of contaminated water.

**Author contribution** All the authors contributed to the study conception and design. Material preparation, data collection, and analysis were performed by Qianyi Zhou, Yan Liu, Shuzhan Wang, Zhen Chen, and Nengzhong Yi. Material characterization and sorption kinetic data analysis were performed by Xing Liu and Zefen Wang. The first draft of the manuscript was written by Rongying Zeng and Wenqing Tang. All the authors commented on previous versions of the manuscript. All the authors read and approved the final manuscript.

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**Data availability** Not applicable.

**Declarations**

**Ethics approval and consent to participate** Not applicable.

**Consent for publication** Not applicable.
Conflict of interest The authors declare no competing interests.

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