A one-pot biosynthesis of an aerogel composite based on attapulgite clay/bacterial cellulose to remove Pb\(^{2+}\) ion

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1 INTRODUCTION

With the industrial development, the wastewater coming from electroplating, electrolysis, medicine, pesticide, and other chemical industries has caused great harms to the ecological environment. This is because the wastewater from industry always contains heavy metal ions, such as Pb\(^{2+}\), Cu\(^{2+}\), Cd\(^{2+}\), which can cause serious environment problems due to their non-degradability and biotoxicity. Many methods have been used to treat heavy metal ions in wastewater, such as chemical precipitation [1], ion exchange [2], membrane [3], and adsorption. Among the above methods, adsorption due to its high removal efficiency and low energy consuming, has been considered as the most affordable and simplest method to removal and recovery of toxic heavy metal ions from wastewater [4]. Furthermore, the adsorbed heavy metals can be collected easily and regenerated by further treatment [5].

Clay minerals are an abundant, low-cost and low or null toxicity resource in nature that have aroused people’s widespread concern [6]. Attapulgite (ATP), a kind of hydrated magnesium-aluminium phyllosilicate clay mineral with nanorod-like crystalline morphology and the theoretical formula of [(Mg, Al)\(_4\)(Si)\(_8\)(O, OH, \(\text{H}_2\)O)\(_{26-n}\)\text{nH}_2\text{O}] [7–9]. Recent years, much attentions have been paid to the application of ATP, owing to its low cost, environment friendly and rich reserves [10]. Due to its unique structure, it owns many physical and chemical properties, such as large specific surface area, good chemical stability, strong adsorption capacity, and slow releasing. Thus it was widely used in water treatment, painting, sensor, and carrier [11–15]. In addition, ATP has been used as a low-cost adsorbant for the removal of heavy metal ions and organic species from wastewater [16]. However, ATP has promising adsorption properties, recovering ATP from wastewater is difficult, energy-consuming and weight loss is unavoidable.

Cellulose is the most abundant natural biopolymer in the earth with good properties such as availability, biocompatibility, biological degradability, and sustainable production [17, 18]. Cellulose can be divided into plant cellulose, bacterial cellulose (BC) and animal cellulose according to its source [19]. In the above three kinds of cellulose, bacterial cellulose produced by some certain bacterial such as *Acetobacter xylinum*, is a novel multifunctional materials [20]. Comparing to other cellulose, it contained no lignin and hemicellulose with high crystallinity [21]. Moreover, due to its high water uptake capacity and its three-dimensional network structure, it has the tendency to form gel [22]. Cellulose aerogels is a lightweight porous solid material have attracted tremendous attention in the respect of multifunctional biopolymer aerogels [23]. As they can feature high specific surface [24], high porosity [25]. It can be used in water treatment, pollutant separation. Some approaches have been tried to treat sewage, such as modified BC membrane with EDTA (Ethylene diamine tetraacetic acid) to adsorb Sr\(^{2+}\) [26]. However, these methods cannot be straightforward to apply on a large scale for they require extra energy consumption or tedious...
steps. Prepared BC composite materials through fermentation in situ in the culture solution has also been carried out to improve the final properties of bacterial cellulose [22]. Anatase TiO$_2$ nanoparticles were incorporated to endow the photocatalytically active to BC [27] and the silica nanoparticles were utilized to improve the elasticity and mechanical strength of BC [28]. Compared with conventional solid adsorbents like ion exchange and chelating resins, main advantages of such materials are easy loading and, in most cases, stripping of cations with simple chemicals, reusability and the possibility of semi-continuous operation. In addition, high wettability and high swelling of hydrogels also might be beneficial for improving adsorption of target metals [27].

In the present work, the ATP and BC composite aerogel were prepared by dynamic air lift fermentation. The prepared method was easy, unlike most cellulose aerogels reported based on dissolution and regeneration of cellulose that need a certain amount of chemicals. This method can disperse attapulgite evenly on cellulose matrix, reduce agglomeration and improve adsorption performance. The ATP/BC composite aerogel was used to study the adsorption behaviour of Pb$^{2+}$. Through this method, the problem of recovering ATP from wastewater is resolved. This work provides a promising approach with low cost and simple procedure to develop an eco-friendly adsorbent, which has a potential prospect in practical application.

2 | MATERIALS AND METHODS

2.1 | Materials

Glucose, sugar, magnesium sulphate, citric acid, ammonium sulphate, potassium dihydrogen phosphate, sodium carboxymethyl cellulose, calcium lactate, acetic acid, corn steep liquor, phosphoric acid, sodium hydroxide, Pb(NO$_3$)$_2$ were all purchased from Sinopharm Group and used as received. Attapulgite was obtained from Xuyi, Jiangsu. Deionized water was used throughout the process. The medium formulation used in the experiment was shown in the Table 1.

2.2 | Methods

2.2.1 | Prepared the ATP/BC aerogel

The media component was weighed according 75 L fermented liquid, and added water to 55 L. (About 10 L of extra water will be produced during the high temperature and high pressure sterilization process, and about 11.5 L of seed liquid in the seed tank will be added to the air lift tank.) and added 15 g (ATP/BC-1), 22.5 g (ATP/BC-2), 30 g (ATP/BC-3) of ATP to the air lift tank respectively, sterilized at 121 °C and 0.12 Mpa, for 20 min. Then cooled down the tank to 30 °C, and about 11.5 L seed liquid from seed tank was added to the air lift tank. Finally, maintained the air lift tank pressure of 0.06 MPa, ventilation volume of 1.7 N·m$^{-1}$·h$^{-1}$, temperature of 30 °C, fermentation production for 60 h. After the fermentation, the obtained flocculent gel clumps was treated with 0.3% NaOH and 0.3% H$_2$O$_2$ at 80 °C for 2 h, repeated several times, until the sample was changed to white. Then rinsed the sample with running water for 24 h. Finally, pre-freeze at −20 °C for 2 h and the sample was freeze-dried at −55 °C for 36 h.

2.2.2 | Characterization

The XRD patterns were obtained by using a Bruker axs d8 advanced diffractometer with Cu Kα radiation ($\lambda = 1.5406 \text{ Å}$) operating at 40 kV and 40 mA with step size of 0.05°. XPS spectra were collected using an RBD-upgraded PHI-5000C ECSA system (PerkinElmer) with Mg K radiation ($h\lambda = 1253.6 \text{ eV}$). The elemental analysis was carried out using a Costech EGS 4010 analyser. Scanning electron microscopy (SEM) images were collected using a Hitachi S-4800 operating at 25 kV to investigate the surface morphologies and internal structures of sample. Before testing, all samples were coated with a thin layer of evaporated gold to enhance the electrical conductivity. The specific surface area and pore size distribution of sample were determined by a surface analyser using the N$_2$ adsorption-desorption method. The specific surface area, pore-volume, and pore diameter were calculated by BET equation and BJH model.

3 | RESULTS AND DISCUSSION

The introduction of ATP in the ATP/BC aerogel had been confirmed by the XPS results. Figure 1 show the elemental analysis of ATP/BC aerogel. The introduction of ATP content in aerogels increased with the amount of ATP added to the culture medium increased during fermentation process. Figure 2 display the XRD patterns of BC and the ATP/BC aerogel. Compared BC and ATP/BC aerogel, they both have peaks at 14.6°, 16.9°, 22.7°, it correspond to the (1 0), (1 1 0), (2 2 0) planes of polymorph cellulose I, respectively [29].

| Reagent                  | Dynamic air lift |
|--------------------------|------------------|
| Glucose                  | 2.25             |
| Sugar                    | 2.75             |
| Magnesium sulphate       | 0.07             |
| Citric acid              | 0.06             |
| Ammonium sulphate        | 0.1              |
| Potassium dihydrogen phosphate | 0.5        |
| Sodium carboxymethyl cellulose | 0.04    |
| Calcium lactate          | 0.02             |
| Acetic acid              | 0.15             |
| Corn steep liquor        | 2                |
| pH                       | 6.0              |

TABLE 1 The medium formulation used in the experimental process (g/100 mL) (The pH was adjusted by using sodium hydrate)
It shows that the introduction of ATP does not change the crystal form of BC and it is still I type of BC. However, it is clear that there is a peak position shift between the BC and ATP/BC aerogel sample around 22.7°, which could attribute to lattice distortion during the ATP and BC combination.

Figure 3 displays the morphologies of BC and ATP/BC aerogel, respectively. From Figure 3(a), we can see the BC aerogel has a three-dimensional network structure and the BC fibres interlace with each other. From Figure 3(b–d), it can be observed that the ATP particles are randomly embedded inside the BC network structure. The dense fibres net can provide good support for the ATP particles, this special structure can effectively prevent the loss of ATP.

The porous properties of BC and BC/ATP aerogel were investigated by N₂ adsorption-desorption experiments. As shown in Figure 4, all samples exhibit type IV isotherms with an H₁ loop, indicating a uniform mesoporous structure. The BET specific surface area of BC, BC/ATP-1, BC/ATP-2 and BC/ATP-3 aerogel are calculated to be 54.2, 80.6, 160.2, 120.4 m²/g respectively. As we can see BC/ATP-2 aerogel is 3.0 times higher than that of pure BC aerogel, suggesting that the introduction of ATP could effectively increase the exposed active surface area for adsorption.

3.1 Adsorption experiments

3.1.1 pH effect

The effect of contact time (0 to 120 min) was also investigated with 0.1 g of composite aerogel and Pb²⁺ initial concentration of 60 mg/L. A series of 60 mg/L Pb²⁺ solutions with different pH (2.0, 3.0, 4.0, 5.0, 6.0) were prepared with acetic acid or sodium hydroxide solutions using a pH meter (DELTA-320).

3.1.2 Adsorption experiments

A series of Pb²⁺ solutions with required concentrations (5, 10, 20, 40, 60, 80, 100, 120 mg/L) were prepared by dissolving Pb(NO₃)₂ in deionized water were followed by subsequent dilutions. Batch adsorption experiments of composite aerogels were conducted with 0.1 g of composite aerogel and 60 mg/L Pb(NO₃)₂ solution at pH = 5.0 and room temperature.

After each completed adsorption reaction, the aerogels were separated by filtration. The initial and equilibrium concentrations were determined by using an atomic absorption spectrometer (AA320ART, Shanghai) in triplicates and the mean value was calculated. The capacities (qₑ) were determined according to Equation (1) as follows:

\[
qₑ = \frac{(C₀ - Cₑ) \times V}{m}
\]

where \(C₀\) (mg/L) and \(Cₑ\) (mg/L) in Equation (1) represent the initial and final equilibrium concentration of absorbate,
respectively; \( V \) (mL) represents the volume of the suspension; \( m \) (g) is the mass of the absorbent.

Figure 5 showed the pH dependence of the adsorption capacity of ATP/BC-2 composite hydrogel to the Pb\(^{2+}\) ion. It is clearly to see that the pH was an important factor affecting the adsorption capacity to Pb\(^{2+}\) of the ATP/BC aerogel. With the pH value increasing, the adsorption capacity of ATP/BC was increase. Which attracted to the media pH value increases from 2 to 5, the H\(^+\) concentration decreases and more free –OH groups are produced, which favours the interaction between –OH and Pb\(^{2+}\) and leads to a significant increase in the adsorption capacity of ATP/BC-2 aerogel to Pb\(^{2+}\). Therefore, the optimum pH value for the adsorption of ATP/BC to Pb\(^{2+}\) was selected to be 5 in order to increase the adsorption efficiency and avoid the hydrolysis of Pb\(^{2+}\) in the neutral medium.

Figure 6 showed the variation of the adsorption capacity to Pb\(^{2+}\) of ATP/BC aerogel with different ATP content. It is clear to see that the adsorption capacity of ATP/BC
FIGURE 5  pH dependence of the adsorption capacity of ATP/BC-2 composite aerogel to the Pb²⁺ ion

FIGURE 6  Variation of the adsorption capacity to Pb²⁺ of BC, ATP and ATP/BC aerogel with different ATP content with contact time

composite hydrogel were high than BC and ATP. In addition, the ATP content was an important factor affecting the adsorption capacity of the composites. As can be seen from Figure 6, under the same conditions, the adsorption capacities of ATP/BC composites with different ATP content are different and the equilibrium adsorption capacity of the composites increased from 25.8 to 34.96 mg/g And the ATP/BC-2 has the best adsorption capacities than other group, which could attributed to ATP/BC-2 has higher ATP content and the ATP was evenly distributed on the bacterial cellulose network, to expose more active sites. Considering the utilization of the raw materials and the adsorption efficiency, ATP/BC-2 was selected as the optimal ATP/BC aerogel adsorbent to further study the adsorption mechanical.

3.1.3  Adsorption kinetics of ATP/BC-2 composite aerogel toward Pb²⁺

To further investigate the adsorption mechanism and its potential rate-controlling steps such as mass transfer, diffusion control and chemical reaction. The adsorption kinetics data are shown in Table 4 and fitted by two different models, that is, the pseudo-first order model (Equation (2)) and the pseudo-second order model (Equation (3)) [30]:

\[ \ln (q_e - q_t) = \ln q_e - k_1 t \]  \hspace{1cm} (2)

\[ \frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2} \]  \hspace{1cm} (3)
where \( q_e \) and \( q_t \) (mg/g) in Equations (2) and (3) are the adsorption capacities of Pb\(^{2+}\) at equilibrium and at time \( t \) (min), respectively. \( k_1 \) (min\(^{-1}\)) and \( k_2 \) (g mg\(^{-1}\) min\(^{-1}\)) are the rate constants of pseudo-first-order and pseudo-second-order, respectively.

Linear plots of pseudo-first-order isotherm (left) and pseudo-second-order isotherm (right) for the adsorption of the Pb\(^{2+}\) ion on ATP/BC-2 aerogel are shown in Figure 7, and the \( R^2 \) calculated from the pseudo-first-order kinetic model and the pseudo-second-order kinetic model were shown in Table 5. As can be seen in Table 5, the values of \( R^2 \) for Pb\(^{2+}\) calculated from the pseudo-second-order kinetic model were found to be over 0.9998, making them higher than those of the pseudo-first-order kinetic model. The results indicated that the adsorption of Pb\(^{2+}\) onto the ATP/BC composite aerogel was well described by the pseudo-second-order kinetic model. It demonstrated that at the beginning of the experiment, the physical absorption on the outer surface of the aerogel seems to be the rate-limiting step. Which attributed to the high specific surface area of ATP and the three-dimensional structure of BC. In addition, chemisorption include ion exchange adsorption was also existing. These different adsorption types, make the adsorption kinetics of the composite aerogel is inclined to pseudo-second-order kinetic model.

The obtained experimental data were analysed with the Langmuir (Equation (4)) and Freundlich (Equation (5)) adsorption isotherms as follows [31]:

\[
\frac{C_e}{q_e} = \frac{1}{q_L \times q_t} + \frac{C_e}{q_t} \quad (4)
\]
\[
\ln q_e = \ln k_f + \frac{1}{n} \ln C_e \quad (5)
\]

where \( q_e \) (mg/g) and \( C_e \) (mg/L) represent the amount of adsorbed Pb\(^{2+}\) per unit mass of ATP/BC composite aerogel and the concentration of the remaining Pb\(^{2+}\) in solution at equilibrium, respectively; \( q_L \) is the maximum monolayer adsorption capacity at a given temperature; \( k_f \) is the equilibrium constant for the adsorption; \( k_f \) and \( n_f \) are system specific constants, \( k_f \) gives an indication for adsorption capacity; \( 1/n_f \) is a measure of intensity of adsorption.

To further investigated the adsorption behaviour of ATP/BC-2 aerogel, the adsorption isotherm drew according to data from Table 6 are shown in Figure 8, the adsorption capacity increased with the equilibrium concentration increased and the increment rate was slow down. The adsorption isotherm fit better to the Freundlich model (0.9868) (Table 3) than Langmuir model with higher \( R^2 \) values, implying that the adsorption
was not as homogeneous as assumed in the Langmuir model, namely, the Freundlich isotherm should be more suitable to evaluate the adsorption here. In other words, the adsorption of Pb\(^{2+}\) on composite aerogel is a kind of adsorption that took place on non-uniform surface.

### 3.1.5 Recyclability of ATP/BC-2 aerogel

The recyclability of the composite aerogel was measured at 25 °C, 0.1 g of composite aerogel was added into 100 mL of 60 mg/L Pb\(^{2+}\) solutions (pH = 5.0) for 90 min, then taken out from the solutions and calculated the adsorption rate. The desorption and regeneration of the Pb\(^{2+}\) loaded composite aerogel were performed by placing these aerogel into 30 mL of 0.1 mol/L hydrochloric acid solution under agitating at 25°C for 30 min. And then washed with deionized water three times, and freeze-dried. The mass of regenerated aerogel was weighed by the analytical balance and then put it into the newly prepared Pb\(^{2+}\) solutions used for another adsorption in the subsequent cycles. The consecutive adsorption–desorption process were performed in triplicate for five times.

The recyclability of aerogel and the removal efficiency of the Pb\(^{2+}\) are important in pollution and environmental protection. As shown in Figure 9, from 1 to 5 cycles, the adsorption rate of aerogel was 96.7%, 93.0%, 91.5%, 89.3% and 88.6%, respectively. After five adsorption-harvesting cycles, the adsorption rate still maintained above 85%. The decreased adsorption rate of aerogel was attributed to the mass loss of aerogel during the desorption and drying. This demonstrated that the aerogel has good recycling ability for the removal of Pb\(^{2+}\).
4 | CONCLUSION

In this work, the ATP/BC composite aerogel were prepared by a one-pot biosynthesis method (dynamic air lift) using eco-friendly materials (ATP) fermentation with Acetobacter xylinum. Compared to pristine ATP, such ATP/BC composite aerogel have the advantages of easy handling and recovery due to their millimetre-size and excellent floatability used for adsorption of Pb\(^{2+}\) in aqueous solution. XPS and SEM analysis had indicated that the existence of ATP in the aerogel. We also have studied the adsorption behaviour of ATP/BC aerogel to Pb\(^{2+}\). The adsorption isotherm and kinetics behaviour was Freundlich, pseudo-second order, respectively. The obtained ATP/BC composite aerogel showed a high adsorption capacity of 34.54 mg/g in 60 mg/L Pb\(^{2+}\) solution within 3 h and after five-cycles the adsorption rate still remained above 85%. The results indicate that the composite aerogel prepared by dynamic air lift culture is a high performance, low-cost, and recyclable green adsorbent.

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