Removal of Cu$^{2+}$ ions from aqueous solution by the abandoned mushroom compost of Flammulina velutipes

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Abstract

The abandoned mushroom compost of Flammulina velutipes, a cheap and easy by-product to get, was used as biosorbent for removing copper ions from aqueous solution. Batch experiments were carried out to investigate the effect of contact time, solution pH, biomass dosage, initial concentration of Cu$^{2+}$ ions and temperature on biosorption efficiency. The maximum sorption capacity could be reached at pH 5.0 in 60 min. Langmuir, Freundlich and Redlich-Peterson isotherm models were used to fit the experimental data and their model parameters were evaluated. The calculated qm based on Langmuir equation was 35.608 mg g$^{-1}$ at 288 K, 48.711 mg g$^{-1}$ at 298 K, and 42.330 mg g$^{-1}$ at 308 K, respectively. The kinetics were discussed by pseudo- first order and pseudo-second order models, and the result showed that the latter was more suitable. The thermodynamics of biosorption was also investigated, and the biosorption process was feasible, spontaneous and endothermic in nature.

Key words

Flammulina velutipes, Cu$^{2+}$ biosorption, Isotherms, Kinetics, Thermodynamics

Introduction

Nowadays, heavy metal pollution has become one of the serious environmental problems, thus threatening the health of human beings by accumulation through food chains. Copper, one of the widely used heavy metals in metallurgy, electroplating, printing and dyeing industry etc has become an environmental pollution. Once the concentration of copper in our blood exceeds a certain value (0.69 to 1.17 mg l$^{-1}$) (Lahey et al., 1953), it may cause several health hazards, such as mucosal irritation and corrosion, capillary damage and central nervous system irritation (Senthil Kumar et al., 2011). Therefore, it is essential to remove copper from aqueous solution.

There are several methods for copper removal, such as chemical precipitation, chemical coagulation, ion exchange, electrochemical methods and adsorption, etc. Most of them have some disadvantages, such as high cost, large energy consumption and complicated disposal process (Farooq et al., 2010). However, adsorption, especially biosorption shows advantages for it is an economical, effective, simple and environmental friendly method. Many investigation have been conducted to identify suitable and relatively cheap biosorbents for copper removal from aqueous solution, like sugar beet pulp (Aksu and Isoglu, 2005), Pleurotus spent mushroom compost (Chia-Chay et al., 2010), Tree fern (Ho, 2003), Boron waste (Olgun and Atar, 2011) and cashew nut sheel (Senthil Kumar et al., 2011).

The demand for Flammulina velutipes in China is relatively large for it being a delicious and welcomed food, which result in large amounts of by-products. The abandoned mushroom compost of Flammulina velutipes, a mixture mainly composed of cottonseed hull and sawdust. The abandoned mushroom compost of Flammulina velutipes is often used as fuel, poultry feeds and fertilizer of flowers, etc. (Qingshang et al., 2005).

The aim of this study is to evaluate the removal efficiency of copper ions by using abandoned mushroom compost of Flammulina velutipes as biosorbent from
simulated wastewater and to investigate the equilibrium, kinetic and thermodynamic characteristics of copper biosorption.

Materials and Methods

Preparation of the biosorbent and reagents: The abandoned mushroom compost of Flammulina velutipes was procured from a Flammulina velutipes workshop, located in Pixian, Chengdu, Sichuan Province of China. The biosorbent was firstly cut into small pieces and air-dried for 48 hr, then ground and sieved through a fine pieces sieve to keep the particles size smaller than 0.3mm, followed by drying at 105°C for 24 hr. The sieved biosorbent was stored in a dry and closed plastic container.

All the chemicals (CuSO$_4$.5H$_2$O, HCl, HNO$_3$ and NaOH) used for the experiments were of analytical grade and purchased from Tianjin Meilin Trade Co. Ltd., China. The double distilled water used throughout was produced by SZ-93A automatic double distilled water rectifier. The solutions of Cu$^{2+}$ were prepared by dissolving accurately weighed amounts of CuSO$_4$.5H$_2$O in double distilled water. The exact concentrations of initial Cu$^{2+}$ or filtrate of Cu$^{2+}$ in the following experiments were determined by the flame atomic absorption photometer (FAAS Z-2300, HITACHI, Japan).

To optimize the biosorption conditions, contact time, biomass dosage, pH and initial concentration of Cu$^{2+}$ in aqueous solution were investigated.

Effect of contact time: To investigate the optimal contact time (or equilibrium time) in biosorption, a series of contact time were conducted: 0, 5, 10, 20, 40, 60, 90, 120, 180, 240, and 360 min. The initial concentration of Cu$^{2+}$ solution was 73.73 mg l$^{-1}$, the pH was kept as nature and biomass dosage was 2.0 g l$^{-1}$. All flasks were sealed and agitated in an incubator at 298 K and 150 rpm. At each set time, about 5 ml of the mixture (its initial volume was 250 ml) was obtained from the origin solution and the biosorbent was separated by filtration. The remained concentration of Cu$^{2+}$ in the filtrate was measured by FAAS.

Effect of biomass dosage: To assess the optimal biomass dosage in biosorption, a series of biomass dosage including 1.0, 2.0, 3.0, 5.0, 8.0, 12.0, 20.0, and 30.0 g l$^{-1}$ were added into Cu$^{2+}$ aqueous solution. The initial concentration of Cu$^{2+}$ solution was 71.80 mg l$^{-1}$, and also kept the pH as nature. All flasks were sealed and agitated in an incubator at 298 K and 150 rpm for 60 min. so that the biosorption could arrive at dynamic equilibrium. Then all the samples were dealt with by the same method as mentioned above.

Effect of pH: To investigate the effect of pH on the biosorption, several pH values, 2.0, 3.0, 4.0, 5.0, 5.5, and 6.0, were experimented. Different volumes of acid (1.0 mol l$^{-1}$ HCl) or alkali (1.0 mol l$^{-1}$ NaOH) were added to the mixture to adjust the pH of the Cu$^{2+}$ solution. The pH values in the solutions were detected with a pH electrode (PHS-25C, Hangzhou Aoilong Instrument Co. Ltd., China). The volume of each solution was 100 ml, with the initial concentration of Cu$^{2+}$ being 68.00 mg l$^{-1}$. The mixtures added with 2.0 g l$^{-1}$ biomass were agitated in the incubator at 298 K for 60 min.. The following procedures were the same as mentioned before.

Effect of initial concentration: To evaluate the effect of initial concentration on the biosorption, the equilibrium isotherms of the biosorption were determined by adding a series of Cu$^{2+}$ solutions ranging from 4.53 to 181.33 mg l$^{-1}$ with 2.0 g l$^{-1}$ biomass into the reaction flasks. The volume of each solution was 100 ml, and kept all the pH values at 5.0. The mixtures were agitated in the incubator at 298K for 60 min.. After that, the following experiments were done with the same methods mentioned above.

The copper uptake q (mg ion metal g$^{-1}$ biomass) was calculated from the mass balance as follows:

$$q = \frac{(C_0 - C_t) \cdot V}{m}$$

(1)

Removal efficiency (%$)$ = \frac{C_0 - C_t}{C_0} \times 100

(2)

where $C_0$ and $C_t$ are the initial and final metal ion concentrations (mg l$^{-1}$), respectively, V is the volume of solution (ml), and m is the biomass weight (g).

Statistical analysis: All treatments were conducted three times and all the data were analyzed with OriginPro 8.5.

Results and Discussion

Time: Fig. 1 shows the effect of contact time on the removal efficiency of Cu$^{2+}$ by the abandoned mushroom compost of Flammulina velutipes. The biosorption of Cu$^{2+}$ increased rapidly in the first few min., and then achieved equilibrium stage. After contacting with the adsorbent for about 60 min., the removal efficiency was above 60%, and then it increased slowly. It was likely that the active binding sites of the biosorbent were occupied by Cu$^{2+}$ ions and achieved saturation phase (Chia-Chay et al., 2010). Based on this result, subsequent experiments were conducted with 60 min of contact time.

Biomass dosage: Considering about economy in treatment, the dosage of biomass is very important when the initial concentration of metal ions in the solution is constant.
Fig. 1 : Effects of contact time on Cu\textsuperscript{2+} removal efficiency with the condition that initial Cu\textsuperscript{2+} concentration of 73.75 mg l\textsuperscript{-1}, pH not adjusted, T=298 K, and r = 150 rpm

Fig. 2 : Effect of biomass on Cu\textsuperscript{2+} biosorption and removal efficiency

Fig. 3 : Lineweaver-Burk plot for determination of half saturation constant on Cu\textsuperscript{2+} biosorption

Fig. 4 : Effect of pH on Cu\textsuperscript{2+} biosorption: The initial concentration was 68.00 mg l\textsuperscript{-1}, biomass of biosorbent was 2.0 g l\textsuperscript{-1}, contact time was 1 hour, T=298K, r=150rpm

\[
\frac{1}{V} = \frac{K_m}{V_{\max}} \frac{1}{[S]} + \frac{1}{V_{\max}}
\]

(4)

where $K_m$ is the half saturation constant of Cu\textsuperscript{2+} biosorption, $V$ (mg (1.min)-1) and $V_{\max}$ (mg (1.min)-1) are the adsorption rate and the maximum adsorption rate of Cu\textsuperscript{2+}, respectively, and [S] (g l\textsuperscript{-1}) is the biomass of the biosorbent.

To save biosorbent dose, this paper took the half of saturation constant of Cu\textsuperscript{2+} biosorption by Michaelis-Menten function:

\[
V = \frac{V_{\max} [S]}{K_m + [S]}
\]

(3)

The above equation can be rearranged to the following linear form (Lineweaver-Burk function):

\[
\text{pH} : \text{pH} \text{ is the most important parameter on biosorption of Cu\textsuperscript{2+}} \text{ from aqueous solutions since it can influence the charges on the biosorbents and the ionic states in the}
\]

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solutions (Chen et al., 2008; Chen et al., 2005; Farooq et al., 2010). As Fig. 4 shows, at low pH value (pH<3), the amount of Cu<sup>2+</sup> absorbed on the biosorbent was low, then increased slightly at the middle pH value (3<pH<5), but increased significantly at high pH value (pH>5). This phenomenon indicated that in spite of many Cu<sup>2+</sup> ions release at lower pH value, the H<sup>+</sup> ions compete with Cu<sup>2+</sup> ions for the exchange sites on the biosorbents by ion exchange or hydrogen bond; and at pH 5 to 6, there were three kinds of copper ions (Cu<sup>2+</sup>, CuOH<sup>+</sup>, and Cu(OH)<sub>2</sub>) in the aqueous system (Elliott and Huang, 1981; Larous et al., 2005), the chemical precipitation is the primary method to remove copper ions from solution. So the most suitable pH value for biosorption on the abandoned mushroom compost of *Flammulina velutipes* is 5.0.

**Initial concentration**: The initial concentration is also an important factor for biosorption to overcome mass transfer resistances of Cu<sup>2+</sup> between the aqueous and biosorbent (Aksu and Isoglu, 2005). The removal efficiency of Cu<sup>2+</sup> in the aqueous solution deceased from 86.54 to 40.99%, while the initial concentration of Cu<sup>2+</sup> increased from 4.53 mg l<sup>-1</sup> to 181.33 mg l<sup>-1</sup>. At lower initial concentration of Cu<sup>2+</sup>, the active exchange sites of biosorbent were not saturated by the copper ions for the mass transfer resistances was very high; and at higher initial concentration of Cu<sup>2+</sup>, the mass transfer resistances decreased relatively, so the copper ions uptaken onto the biosorbents increased (Chia-Chay et al., 2010). But the availability of active binding sites of biosorbents decreased relatively when the initial concentration of Cu<sup>2+</sup> increased, which result in the decrease of removal efficiency of Cu<sup>2+</sup> in the solution (Meena et al., 2008).

**Sorption isotherms**: Three common adsorption equilibrium models, namely Langmuir, Freundlich, and Dubinin-Radushkevich, shown in Table 1, were used to fit the experimental data for Cu<sup>2+</sup> biosorption equilibrium on the abandoned mushroom compost of *Flammulina velutipes* at different temperatures (288, 298 and 308 K).
Table 1: Original and linearized forms of three biosorption isotherm models

<table>
<thead>
<tr>
<th>Isotherm model</th>
<th>Original equation</th>
<th>Linearized equation</th>
<th>Graphical dependence</th>
</tr>
</thead>
<tbody>
<tr>
<td>Langmuir model</td>
<td>$q_e = q_m \frac{k_e \cdot C_e}{1 + k_e \cdot C_e}$</td>
<td>$C_e = \frac{1}{K_a q_m} + \frac{1}{q_m} \cdot C_e$</td>
<td>$C_e$ vs. $C_e$</td>
</tr>
<tr>
<td>Freundlich model</td>
<td>$q_e = K_f \cdot C_e^{1/n}$</td>
<td>$\log q_e = \log K_f + \frac{1}{n} \log C_e$</td>
<td>$\log q_e$ vs. $\log C_e$</td>
</tr>
<tr>
<td>Redlich-Peterson model</td>
<td>$q_e = \frac{A C_e}{1 + BC_e}$</td>
<td>$\ln \left( \frac{A}{q_e} - 1 \right) = g \ln C_e + \ln B$</td>
<td>$\ln \left( \frac{A C_e}{q_e} - 1 \right)$ vs. $\ln C_e$</td>
</tr>
</tbody>
</table>

Notation: $q$ is the equilibrium metal ion concentration on the adsorbent (mg g⁻¹), $C_e$ is the equilibrium metal ion concentration in the solution (mg l⁻¹), $q_m$ is the maximum adsorption capacity on the monolayer of adsorbent (mg g⁻¹), $K_f$ is sorption equilibrium constant (L mg⁻¹), $K_f$ is Freundlich constant, $1/n$ is the heterogeneity factor, $A$, $B$, and $g$ are Redlich-Peterson constant.

Table 2: The value of parameters for each isotherm models used in this study

<table>
<thead>
<tr>
<th>Temperature (K)</th>
<th>Langmuir constants</th>
<th>Freundlich constants</th>
<th>Redlich-Peterson constants</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$K_s$</td>
<td>$q_m$</td>
<td>$R^2$</td>
</tr>
<tr>
<td>288</td>
<td>0.092</td>
<td>35.608</td>
<td>0.9943</td>
</tr>
<tr>
<td>298</td>
<td>0.034</td>
<td>48.711</td>
<td>0.9885</td>
</tr>
<tr>
<td>308</td>
<td>0.053</td>
<td>42.330</td>
<td>0.9887</td>
</tr>
</tbody>
</table>

Table 3: A number of agricultural by-products available for biosorption of copper ions

<table>
<thead>
<tr>
<th>Material</th>
<th>$q_m$ (mg g⁻¹)</th>
<th>Contact time (min)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cashew nut shell</td>
<td>20.0</td>
<td>30</td>
<td>(Senthil Kumar et al., 2011)</td>
</tr>
<tr>
<td>Pleurotus ostreatus spent mushroom compost</td>
<td>4.6</td>
<td>10</td>
<td>(Chia-Chay et al., 2010)</td>
</tr>
<tr>
<td>Sawdust</td>
<td>8.5</td>
<td>5</td>
<td>(Larous et al., 2005)</td>
</tr>
<tr>
<td>Sugar beet pulp</td>
<td>31.4</td>
<td>60</td>
<td>(Aksu and Isoglu, 2005)</td>
</tr>
<tr>
<td>Tree fern</td>
<td>11.7</td>
<td>300</td>
<td>(Ho, 2003)</td>
</tr>
<tr>
<td>Abandoned Mushroom Compost of Flammulina velutipes</td>
<td>48.7</td>
<td>60</td>
<td>This study</td>
</tr>
</tbody>
</table>

The Langmuir sorption isotherm is based on the assumption that sorption takes place at specific homogeneous surfaces on the sorbent, where interaction between adsorbed molecules are negligible (Langmuir, 1918). The Freundlich model assumes a heterogeneous adsorption surface and applies well when the adsorption takes place in aqueous systems (Freundlich, 1906). The Redlich-Peterson isotherm model incorporates the features of the Langmuir and the Freundlich isotherms (Redlich and Peterson, 1959).

In order to assess different adsorption isotherms and their ability to fit with experimental data, the Fig.6 shows each isotherm models of copper ions adsorbed on the adsorbents at 298 K. All the parameters of three isotherm models have been summarized in Table 2. The coefficients of determination, $R^2$, are greater than 0.988 for the Langmuir and the Redlich-Peterson isotherms, but lower than 0.967 for the Freundlich isotherm at different temperatures, respectively. So, it indicated that the Langmuir and the Redlich-Peterson isotherms had fitted the experimental data better than Freundlich at various temperatures, obviously.

The calculated Langmuir constants $K_s$ and $q_m$ have also been listed in Table 2. The $q_m$ was found to increase...
Table 4: Kinetic parameters obtained from pseudo-first order and pseudo-second order for Cu²⁺ biosorption onto the substrate of Flammulina velutipes at different temperatures.

<table>
<thead>
<tr>
<th>Temperature (K)</th>
<th>Pseudo-first order</th>
<th>Pseudo-second order</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$q_{e,exp}$ (mg g⁻¹)</td>
<td>$k_1$ (min⁻¹)</td>
</tr>
<tr>
<td>288</td>
<td>19.70</td>
<td>$5.26 \times 10^{-2}$</td>
</tr>
<tr>
<td>298</td>
<td>19.73</td>
<td>$7.10 \times 10^{-2}$</td>
</tr>
<tr>
<td>308</td>
<td>18.87</td>
<td>$1.58 \times 10^{-1}$</td>
</tr>
</tbody>
</table>

from 35.608 to 48.711 mg g⁻¹, and then decreased to 42.330 mg g⁻¹ with an increase in the solution temperatures from 288 to 308 K. In addition, the values of $K_e$ decreased at first stage, and then increased as the solution temperature increasing. The values of constants A and B of Redlich-Peterson isotherm had the similar regulars with the solution temperature increased. In order to compare the value of maximum sorption capacity ($q_{max}$) of present study to the former works of other researchers, a number of agricultural by-products available for biosorption of copper ions were listed in Table 3. Obviously, the abandoned mushroom compost of Flammulina velutipes has a higher adsorption capacity than other adsorbents, but a longer contact time when compared with sawdust, Pleurotus ostreatus spent mushroom compost and cashew nut shell.

The parameters of Langmuir isotherm can be used to predict the affinity efficiency in the biosorption system. The essential characteristics of the Langmuir isotherm can be expressed by using separation factor or dimensionless equilibrium parameter, $K_R$, as following equation:

$$K_R = \frac{1}{1 + K_e C_0}$$

Where $K_R$ is the Langmuir constant, 1 mg⁻¹, and $C_0$ is the initial concentration of Cu²⁺, mg l⁻¹. The value of $K_R$ indicated the type of Langmuir isotherm to be irreversible ($K_R = 0$), favourable ($0 < K_R < 1$), linear ($K_R = 1$), or unfavourable ($K_R > 1$). The calculated values of $K_R$ was found to be 0.063 to 0.730 for concentration of 4.03-161.17 mg l⁻¹ of Cu²⁺ at 288 K, 0.140 to 0.866 for concentration of 4.53-181.33 mg l⁻¹ of Cu²⁺ at 298 K, 0.105 to 0.824 for concentration of 4.03-161.17 mg l⁻¹ of Cu²⁺ at 308 K.

Kinetics of sorption: The biosorption rate is very important when designing batch sorption systems (Sari and Tuzen, 2009). In order to investigate the biosorption kinetics of Cu²⁺ onto the substrate of Flammulina velutipes, two kinetics models, pseudo-first order and pseudo-second order models, were applied to the kinetics experimental data.

The linear forms of the pseudo-first order and pseudo-second order kinetics models can be written as follows:

$$\ln(q_t - q_e) = \ln(q_{e,exp} - k_1 \cdot t)$$

$$\frac{t}{q_t} = \frac{1}{k_2 \cdot q_{e,exp}^2} + \frac{t}{q_{e,exp}}$$

where $q_t$ is the amount of heavy metal ions adsorbed at equilibrium and at time $t$ (min), respectively, $k_1$ is the rate constant of the pseudo-first order kinetics equation (min⁻¹), and $k_2$ is the pseudo-second order rate constant (g mg⁻¹ min⁻¹).

The biosorption rate constants $k_1$ can be calculated by plotting of ln($q_t - q_e$) versus $t$ at 288-308 K. Because of the coefficients of determination for this model is very low, the figure was not shown here. From the $R^2$ values in Table 4, it is easy to judge that the biosorption mechanisms of Cu²⁺ onto the substrate of Flammulina velutipes does not fit the pseudo-first order kinetic model. What’s more, the calculated values of $q_{t=exp}$ are quite different with the experimental values of $q_{t=exp}$ as shown in Table 4. Therefore, the pseudo-first order kinetic model is not suitable for this system.

The pseudo-second order kinetic model is based on the assumption that the rate-controlling step in the biosorption process is chemical interactions between functional groups of biosorbent and heavy metal ions (Bulgariu and Bulgariu, 2011). The rate constants $k_2$ of biosorption can be calculated by plotting of $t/q_t$ versus $t$ at 288-308 K, as shown in Fig. 7. The rate constants $k_2$ obtained the $q_{t=exp}$ and the coefficients $R^2$ are summarized in Table 4, too. It is obvious that the values of $R^2$ for the pseudo-second order kinetic model are very high (in range of 0.9997-0.9999). In addition, the calculated values of $q_{t=exp}$ are very close to the experimental values $q_{t=exp}$. In the view of these results, it indicated that the pseudo-second order kinetic model is better in describing the biosorption kinetics of Cu²⁺ onto the substrate of Flammulina velutipes.

Thermodynamics of sorption: The theory of thermodynamics assumed that the entropy change is the driving force in an isolated system without any energy.
exchange with circumstances (Ho, 2003). In order to describe thermodynamic behavior of the biosorption of Cu\(^{2+}\) onto the substrate of Flammulina velutipes, thermodynamic parameters including the Gibbs free energy (\(\Delta G^0\)), enthalpy (\(\Delta H^0\)) and entropy (\(\Delta S^0\)) were studied in this work. The relationship between free energy of sorption reaction, and the sorption equilibrium constant, \(K_s\), is given by the following equation:

\[
\Delta G^0 = -RT \ln K_s
\]

(8)

Where \(\Delta G^0\) is standard free energy change, J; \(R\) is the universal gas constant, 8.314 J (mol\(^{-1}\) K\(^{-1}\)); \(T\) is absolute temperature, K; \(K_s\) is the distribution coefficient (\(q/C\)), l g\(^{-1}\).

The enthalpy (\(\Delta H^0\)) and entropy (\(\Delta S^0\)) parameters were estimated from the following equation:

\[
\ln K_s = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT}
\]

(9)

According to Eq. (8) and (9), the Gibbs free energy change (\(\Delta G^0\)) can be represented as following equation:

\[
\Delta G^0 = \Delta H^0 - T \Delta S^0
\]

(10)

A plot of Gibbs free energy change (\(\Delta G^0\)) versus temperature, T, was found to be linear (the R\(^2\) was 0.984). The parameters \(\Delta H^0\) and \(\Delta S^0\) could be calculated from the intercept and slope of the fitting line, as showed in Fig. 8. The \(\Delta G^0\) was calculated to be -6.86, -6.95, -7.05, -7.18, -7.36, -7.44 and -7.62 kJ mol\(^{-1}\) for biosorption of Cu\(^{2+}\) at 278, 283, 288, 293, 298, 303 and 308 K, respectively. The \(\Delta H^0\) and \(\Delta S^0\) for biosorption were calculated to be 0.146 kJ mol\(^{-1}\) and 0.025 kJ mol(K)\(^{-1}\), respectively. The negative values of \(\Delta G^0\) indicated thermodynamically was feasible and spontaneous nature of the biosorption. The positive value of \(\Delta H^0\) indicated that the sorption reaction is endothermic. The positive value of \(\Delta S^0\) reflected the increasing in the randomness at the solid/solution interface during the biosorption process. Özcan, et al. (2005) reported similar observation by using seeds of Capsicum annuum as biosorbent for Cu\(^{2+}\) adsorption.

The results indicated that the Langmuir and Redlich-Peterson isotherm models well fitted the sorption of Cu\(^{2+}\) at different temperatures, the kinetics of biosorption follows the pseudo- second order model, and the biosorption is a feasible and spontaneous endothermic reaction in nature. Study shows that the abandoned mushroom compost of Flammulina velutipes has the ability to be a good material for heavy metal bioremediation.

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