

Removal of phenylalanine from water with calcined CuZnAl–CO₃ layered double hydroxides

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Received 5 January 2011; accepted 1 August 2011

Abstract: Cu–Zn–Al–CO₃ layered double hydroxide (LDH), with a Cu to Zn mole ratio of 5:1 and a (Cu+Zn) to Al mole ratio of nearly 2, was prepared and its calcined product (CLDH) was obtained. Batch sorption studies were conducted to investigate removal of phenylalanine from water with CLDH. The results show that CLDH can be used as an effective adsorbent and its sorption capacity is higher than that of Mg–Al–CO₃–LDH. The maximum adsorption was observed at pH 6.7. A maximum adsorption capacity is 37.25 mg/g. The adsorption processes follow the Lagergren's first order kinetic model. The adsorption data are fitted well with the Langmuir isotherm equation. The thermodynamic parameters were calculated, and the negative ΔG^\ominus and positive ΔH^\ominus indicate that the adsorption processes are spontaneous endothermic in nature. The mechanism of adsorption also suggests that the benzoate molecules are tilted, forming an angle with the hydroxyl layers.

Key words: Cu–Zn–Al–CO₃–LDH; phenylalanine; adsorption; mechanism

1 Introduction

Phenylalanine (Phe) is an amino acid that is widely used in many fields, such as food, chemical, pharmaceutical, agricultural, medicine, and cosmetic industries [1]. To improve the efficiency of recovery, separation and purification of amino acids from wastewater coming from fermentations broths, several techniques have been employed [2–5]. In this way, many studies have been reported for adsorption of amino acids on various materials including activated carbon, silica, ion exchangers, alumina, and polymeric resins [6–10]. A class of anionic clays known as layered double hydroxides (LDHs) has been proved to be effective adsorbents for removal of a variety of anionic pollutants. The general formula of LDHs is $[M^{II}]_{1-x}M^{III}_x(OH)_2(A^{n-})_{x/n} \cdot mH_2O$, where M^{II} is a divalent metal cation (Mg²⁺, Zn²⁺, Cu²⁺, etc), and M^{III} is a trivalent metal cation (Al³⁺, Cr³⁺, Fe³⁺, etc) that occupies octahedral sites in the hydroxide layers, Aⁿ⁻ is a

exchangeable interlayer anion, and x is defined as the ratio of M^{II} to (M^{II}+M^{III}) on which the layer charge will depend. Calcined layered double hydroxide (CLDH) is capable of recovering the LDH layered structure upon treatment with water or aqueous solution containing various anions via the so called "memory effect", and has been demonstrated to be good ion exchangers/adsorbents for removal of toxic anions from contaminated water [11,12].

In this study, Cu–Zn–Al–CO₃–LDH was synthesized, calcined, and then used for removal of Phe from water. The effects of several conditions on the removal process including the pH value of aqueous solution, the temperature, Phe–adsorbent contact time, and the Phe removal process have been investigated. A comparative experiment was conducted on Mg–Al–CO₃–LDH. The equilibrium isotherm and kinetic model for the adsorption processes were studied to evaluate the viability and effectiveness of the process. The adsorption mechanism was also discussed with the supports of XRD, FT-IR results.

Foundation item: Project (21176263) supported by the National Natural Science Foundation of China; Project (2009RS3039) supported by Hunan Provincial Postdoctoral Special Foundation of China; Project (09JJ3026) supported by Hunan Provincial Natural Science Foundation of China

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DOI: 10.1016/S1003-6326(11)61201-6

2 Experimental

2.1 Preparation of Cu–Zn–Al–CO₃–CLDH

A solution containing Na₂CO₃ (0.09 mol/L) and NaOH (0.36 mol/L) was added to 500 mL distilled water. Another solution containing a mixture of metal salts of Cu(NO₃)₂·3H₂O (0.10 mol/L), Zn(NO₃)₂·6H₂O (0.02 mol/L) and Al(NO₃)₃·9H₂O (0.06 mol/L) (Cu to Zn mole ratio of 5:1 and a Cu+Zn to Al mole ratio of nearly 2.0) was prepared, and it was added dropwise to the alkaline solution with continuous mechanical stirring. The pH was adjusted to 10 by the simultaneous addition of the mixture of Na₂CO₃ and NaOH. Once addition was completed, the slurry was mechanically stirred at room temperature for 1 h. The material obtained was submitted to hydrothermal reaction vessel treatment at 80 °C for 6 h. The slurry was centrifuged and washed several times with warm deionized water until neutral pH was reached [13]. CLDH was obtained by calcining LDH at 500 °C for 12 h.

2.2 Instrumentation

The structural characterizations were performed by X-ray diffraction (XRD) and IR spectroscopy. Powder XRD measurements were performed on a Rigaku Rint 6000 powder X-ray diffractometer, using Cu K_α radiation at 30 mA, 40 kV. The IR spectra were obtained on a NICOLET AVATAR 360 spectrophotometer by the standard KBr disk method. A temperature-controlled shaker bath with a temperature variation of ±0.5 °C was used for the equilibrium studies.

2.3 Reagents

Phe was used without further purification. Ninhydrin solution was obtained by dissolving 5.0 g Na₂HPO₄·12H₂O, 3.0 g KH₂PO₄, 0.25 g ninhydrin and 0.15 g fructose in 50 mL volumetric flask (pH= 6.7). The stabilizer potassium iodate was obtained by dissolving 1.0 g KIO₃ in mixture of 300 mL distilled water and 200 mL 95% ethanol.

2.4 Batch adsorption experiments

The adsorption experiments were carried out in 100 mL stoppered conical flasks by mixing a 50.0 mL Phe solution with appropriate concentration and 0.10 g CLDH and shaking immediately in a constant temperature water bath. The mixtures were filtered at given time intervals. The experiments were carried out at different temperatures and repeated two times. The initial pH values of Phe working solution were adjusted by addition of HCl or NaOH solution.

2.5 Detection method

The concentration of Phe was detected by ninhydrin colorimetry on a UV-Vis spectrophotometer (UV-9600) by measuring absorbance at 569 nm. The supernatants were adjusted to 6.7 by addition of HCl or NaOH solution before detection. 2.0 mL Phe solution and 0.5 mL ninhydrin solution were mixed in a 25 mL color comparison tube. After heating for 16 min in boiling water and cooling for 15 min, 5.0 mL potassium iodate was added to the mixture. The mixture was detected within 30 min.

The removal efficiency was measured by the removal rate (*r*) of Phe:

$$r = \frac{\rho_0 - \rho_e}{\rho_0} \times 100\% \quad (1)$$

where ρ_0 and ρ_e are the initial concentration and equilibrium concentration of the Phe in solution (mg/L), respectively.

3 Results and discussion

3.1 Characterization of materials

The LDH and CLDH after adsorbing Phe (PLDH) were characterized by XRD. Figure 1(a) shows the diffraction pattern of LDH. The XRD patterns present sharp, symmetric, strong lines at low 2θ . It is characteristic of well-crystallized product with layered structure of LDH at d_{003} (0.762 nm) and d_{006} (0.379 nm). Figure 1(b) shows the pattern of CLDH. The layered structure collapses due to the loss of interlayer water molecules, carbonate anions as carbon dioxide and surface hydroxyl groups. The characteristic reflections of a mixed oxides CuAl(O), CuO and ZnO appear at 2θ values of 31.6° and 36.8°, 35.4°, 38.7° and 32.4°, respectively. In Fig. 1(c) the diffraction pattern of PLDH is present. The reappearance of characteristic layered structure of LDHs indicates that the adsorption of Phe is by the ‘memory effect’ of CLDH. A new

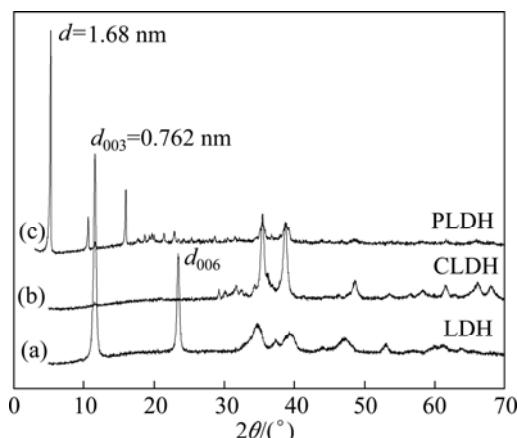


Fig. 1 XRD patterns of LDH (a), CLDH (b) and PLDH (c)

diffraction pattern appears at 5.35° . The increase in peak intensity and the reduction in peak width after intercalation of Phe indicate an increase in crystallization.

The IR spectrum of LDH (Fig. 2(a)) resembles those of other hydrotalcite-like phases [13]. Typical spectra are the strong broad absorbance bands between 3600 and 3200 cm^{-1} associated with the stretching mode of hydrogen-bonded hydroxyl groups from both the hydroxide layers and interlayer water. The strong asymmetric stretching mode of the carbonate ions is found at 1391 cm^{-1} . The IR spectrum of Phe is shown in Fig. 2(c). A series of bands are recorded at 1074 , 700 , 1562 and 1625 cm^{-1} ascribed to vibration peaks of phenyl ring groups. The characteristic absorption peaks of carbonyl group are recorded at 1409 , 1456 cm^{-1} , and the bands due to NH_3^+ are recorded at 849 , 3066 cm^{-1} . The IR spectra of Phe (Fig. 2(b)) were preserved in PLDH, but the intensity decreases. The band due to NH_4^+ appears at 849 cm^{-1} . The intensity of vibration peaks of aromatic ring recorded at 1076 , 698 , 1560 and 1619 cm^{-1} and carbonyl group recorded at 1456 , 1409 , 949 cm^{-1} decreased or disappeared in PLDH. It is shown that the Phe was adsorbed into interlayer and there is an interaction between Phe and LDH layers.

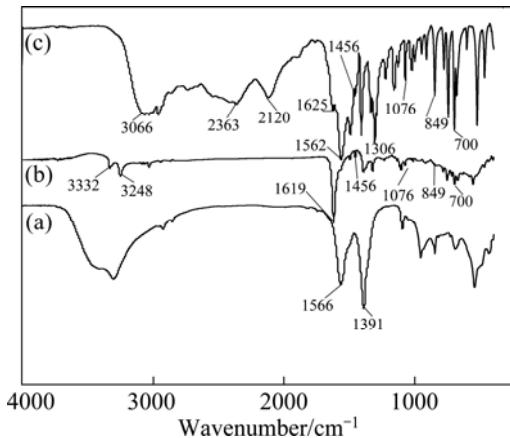


Fig. 2 IR spectra of LDH (a), PLDH(b) and Phe (c)

3.2 Effect of pH

Generally, pH is considered to be an important factor to control the adsorption at water–adsorbent interfaces [14]. So, the adsorption of Phe (0.1 g/L) on CLDH (0.1 g) was studied at different pH values ranging from 5 to 9 .

The influence of pH on the Phe sorption is presented in Fig. 3. Near isoelectric point ($\text{pH}=5.48$), the decrease of the adsorption can be attributed to the zero net charge of Phe. On the other hand, for $\text{pH}>7$, carbonate contamination is likely to occur and may hinder the Phe adsorption due to the great affinity of LDH for carbonate anions [15]. The CLDH recorded the

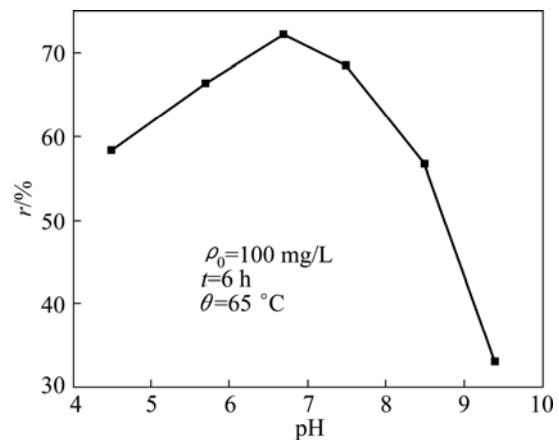
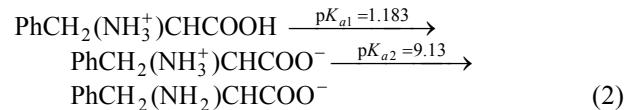


Fig. 3 Effect of pH on Phe retention by CLDH

maximum adsorption at pH 6.7 . Hence, the adsorption experiments were carried out at pH 6.7 .



where Ph represents phenyl ring.

3.3 Temperature effect

The effect was studied on suspensions of CLDH in Phe solutions (100 mg/L Phe concentration; 1.0 g/L solid/solution ratio) stirred at three constant temperatures (313 , 338 and 353 K), respectively. Suspensions were centrifuged; the supernatants were adjusted to pH 6.7 and the Phe concentrations were determined as above.

As the adsorption process is strongly dependent upon temperature [16], the study of the temperature effect on Phe removal with CLDH enabled us to determine the thermodynamic parameters (ΔG^\ominus , ΔH^\ominus and ΔS^\ominus) of these reactions by using the following equations:

$$\Delta G^\ominus = -RT \ln K_d \quad (3)$$

$$\ln K_d = \frac{\Delta S^\ominus}{R} - \frac{\Delta H^\ominus}{RT} \quad (4)$$

where R is the gas constant; T is the temperature, K; K_d is the distribution coefficient (amount of removed Phe per gram of material divided by its concentration in the liquid phase). The plot of $\ln K_d$ against T^{-1} gives a straight line, and the slope and the intercept correspond to $\Delta H^\ominus/R$ and $\Delta S^\ominus/R$, respectively.

The plot of $\ln K_d$ vs T^{-1} for Phe removal with CLDH (Fig. 4) shows a negative slope. The thermodynamic parameters calculated from the values of the slopes and the intercepts are listed in Table 1. The negative value of ΔG^\ominus and positive value of ΔH^\ominus indicate that the adsorption processes are spontaneous and endothermic in nature. ΔS^\ominus can be used to describe the randomness at

the solid–solution interface during the removal process. The positive value of ΔS^Θ reflects the affinity of CLDH to the Phe ion in aqueous medium. The process may be masked by the increased intraparticle diffusion rate of Phe into the pores of LDH at higher temperatures that give rise to the observed increase in their adsorption.

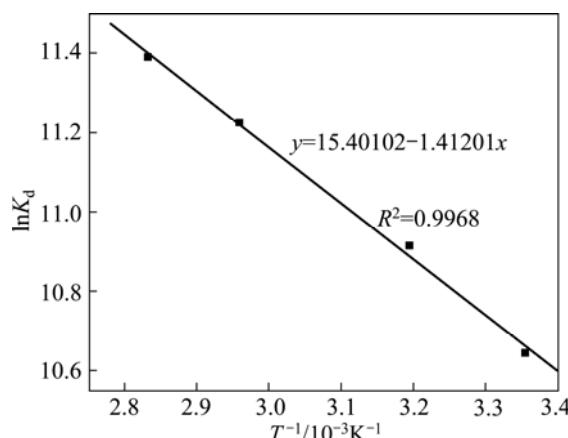


Fig. 4 Plot of $\ln K_d$ vs T^{-1} for Phe removal with CLDH

Table 1 Thermodynamic parameters for Phe removal with CLDH

$\Delta S^\Theta / (\text{J}\cdot\text{mg}^{-1}\cdot\text{K}^{-1})$	$\Delta H^\Theta / (\text{kJ}\cdot\text{mg}^{-1})$	$\Delta G^\Theta / (\text{kJ}\cdot\text{mg}^{-1})$		
		313 K	338 K	353 K
125.86	10.986	-28.41	-31.55	-33.44

3.4 Effect of contact time

The amount of Phe adsorbed by CLDH as a function of contact time, using a constant adsorbent mass of 100 mg and initial concentration of 100 mg/L, is shown in Fig. 5. The kinetic curve of the Phe adsorption on the CLDH is achieved at 50 °C. It can be seen from Fig. 5 that the adsorption increases initially and then reaches the equilibrium state after a contact time of 10 h in the case of no change in the adsorbed amount. The similar phenomenon was observed by EI GAINI et al [14]. A comparative experiment was conducted on Mg–Al–CO₃–LDH. The result showed that the Cu–Zn–Al–CO₃–CLDH is a better adsorbent for removal of Phe than Mg–Al–CO₃–LDH. From these experiments, we also find that Cu–Zn–Al–CO₃ CLDH is a better choice for adsorption.

3.5 Adsorption kinetic

The sorption kinetic is an important aspect of processes for pollutants removal [17–21]. In order to investigate the adsorption mechanism of Phe on CLDH, the Lagergren's first order kinetic model and the Ho's pseudo-second-order model were applied to describing the experimental process.

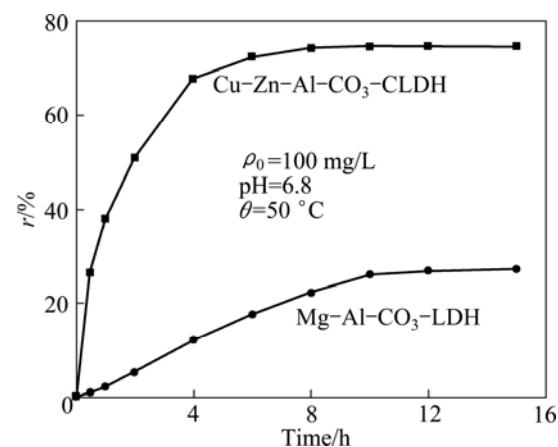


Fig. 5 Curves of Phe removal with CLDH

The Lagergren's first order kinetic model [21] is generally expressed in integrated form as follows:

$$\lg(Q_e - Q_t) = \lg Q_e - \frac{k_1}{2.303} t \quad (5)$$

where k_1 is the rate constant of adsorption, h^{-1} ; Q_e and Q_t (mg/g) are the adsorption capacities of the adsorbate at equilibrium and at time t (h), respectively. By plotting $\lg(Q_e - Q_t)$ against t , a linear graph can be obtained and the value of k_1 and Q_e can be calculated. The result is plotted in Fig. 6 and the calculated parameters are $k_1 = 0.6138 \text{ h}^{-1}$, $Q_e = 36.37 \text{ mg/g}$.

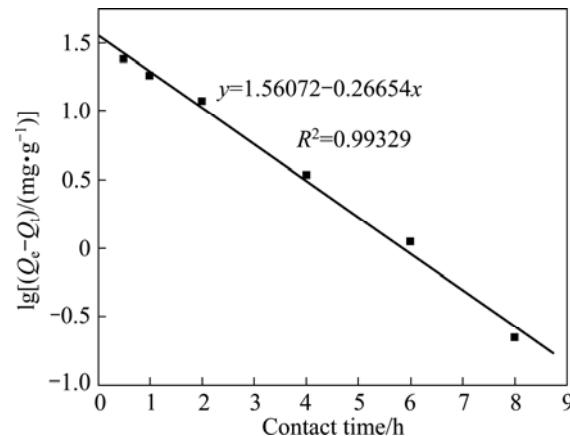


Fig. 6 First order plot for Phe removal with CLDH

Due to its good correlation with the experimental results, the more recent pseudo-second-order model has been extensively used by several researchers in the same field [21]. This model is expressed as

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{t}{Q_e} \quad (6)$$

where k_2 is the rate constant of pseudo-second-order adsorption, $\text{g}/(\text{mg}\cdot\text{h})$. By plotting t/Q_t against t , a linear function can be obtained and the rate constant k_2 as well

as Q_e also can be calculated. The result is plotted in Fig. 7 and the calculated parameters are $k_2=0.01916$ g/(mg·h), $Q_e=43.01$ mg/g, respectively.

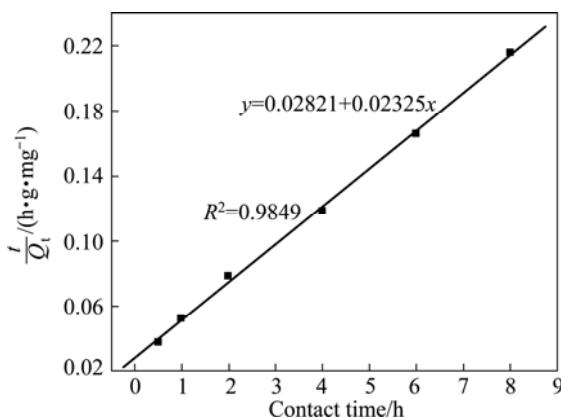


Fig. 7 Pseudo second order plot for Phe removal with CLDH

Good correlation is observed between experimental data and the Lagergren's first order kinetic model with a determination coefficient value higher than 0.99. This indicates that the process of Phe removal is controlled by diffusion rather than by the rate of reaction of Phe with CLDH.

3.6 Adsorption isotherms

The equilibrium adsorption experimental data obtained in this study were analyzed using the commonly used Freundlich and Langmuir isotherm models. The Freundlich isotherm model is described by the following equation:

$$Q_e = K_F \rho_e^{1/n} \quad (7)$$

where Q_e is the equilibrium sorption concentration of solute per gram of adsorbent; mg/g; ρ_e is the equilibrium aqueous concentration of the solute, mg/L; K_F and n are Freundlich constants which are related to adsorption capacity and intensity of adsorption.

The result is plotted in Fig. 8 and the calculated

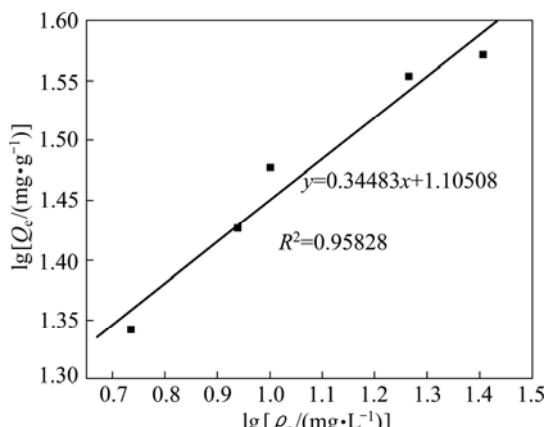


Fig. 8 Freundlich isotherm for Phe removal with CLDH

parameters are $K_F=12.74$, $n=2.90$, $R^2=0.95828$.

The Langmuir isotherms model is described by the following equation:

$$Q_e = \frac{Q_{\max} K_L \rho_e}{1 + K_L \rho_e} \quad (8)$$

where Q_e is the amount of Phe removed per gram of adsorbent, mg/g; Q_{\max} is the maximum sorption capacity, mg/g; ρ_e is the Phe concentration in the equilibrium solution, mg/L; K_L is the Langmuir constant related to the adsorption energy, L/mg.

The result is plotted in Fig. 9 and the calculated parameters are $Q_{\max}=46.4$ mg/g, $K_L=0.168$ L/mg, $R^2=0.99722$.

The experimental result is well described by both models, but the Langmuir model remains better. It may be due to the homogenous distribution of active sites on the CLDH surface.

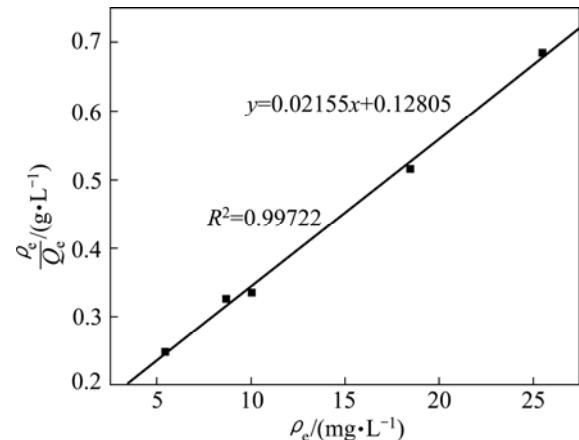


Fig. 9 Langmuir isotherm for Phe removal with CLDH

3.7 Mechanism of adsorption of Phe

Based on the results obtained by IR spectra and XRD, we can deduce that the adsorption of Phe is carried out by reconstruction of a matrix hydrotalcite intercalated by the Phe bounding in the interlayer spaces through electrostatic interactions and hydrogen bonding. The process takes place by reconstruction of oxide, with interrelation of hydroxyl anions, which are subsequently replaced by Phe via an ion-exchange process. The thickness of the LDHs layer is 0.48 nm, and the gallery height of $[\text{CuZnAl}-\text{CO}_3]$ after retention is 1.20 nm. The value of long axis of Phe anion (0.745 nm), calculated from the semi-empirical molecular orbital method of Gaussian 03 software, is well consisted with the gallery height of $[\text{CuZnAl}-\text{CO}_3]$ after the uptake of Phe anion, as shown in Fig. 10. NEXAFS studies by MOGGRIDGE et al [22] indicated a bilayer orientation, but the benzoate molecules are tilted, forming an angle of $(35 \pm 10)^\circ$ with the hydroxyl layers.

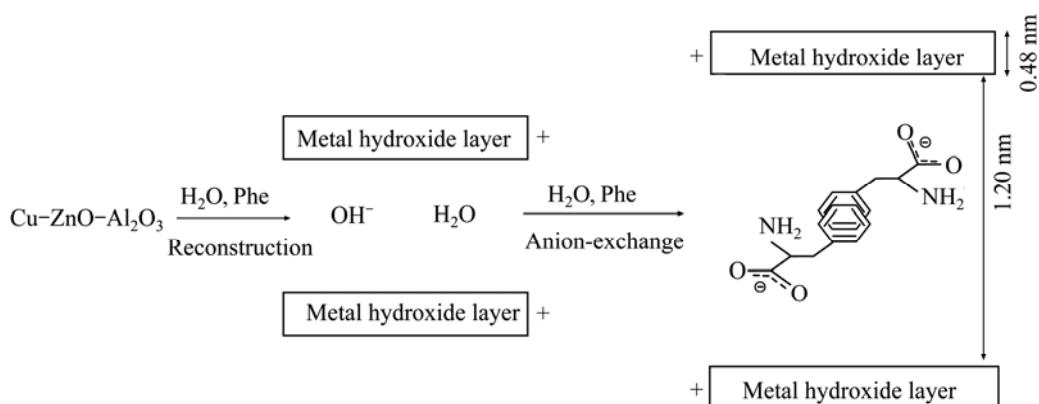


Fig. 10 Schematic illustration of Phe intercalation into layer

4 Conclusions

1) It was confirmed that CLDH might be a potential material for the removal of Phe from aqueous solution. The sorption was found to be a pH dependent and the maximum adsorption was observed at pH 6.7. A maximum adsorption capacity is 37.25 mg/g.

2) The kinetic and sorption data fit well the Lagergren's first order kinetic model and the Langmuir model respectively with good values of the determination coefficient. The negative value of ΔG^\ominus and the positive value of ΔH^\ominus indicate that the adsorption processes are spontaneous endothermic in nature.

3) The mechanism of adsorption also suggests that the benzoate molecules are tilted, forming an angle with the hydroxyl layers.

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焙烧型 CuZnAl 双金属氢氧化物对 水中苯丙氨酸的脱除

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摘要: 用水热法合成了 Cu-Zn-Al-CO₃ 双金属氢氧化物 $n(\text{Cu})/n(\text{Zn})=5$, $[n(\text{Cu})+n(\text{Zn})]/n(\text{Al})\approx 2$, 简写为 LDH, 加热煅烧后得到焙烧型 Cu-Zn-Al-CO₃-LDH(CLDH), 再采用批处理实验用其对模拟废水中的苯丙氨酸进行吸附。结果表明, CLDH 对于废水中的苯丙氨酸是一种有效的吸附剂, 其吸附效率明显高于同等条件下的 LDH, 当水相 pH 为 6.7 时, 其吸附效率最高, 吸附容量达到 37.25 mg/g。该吸附过程符合 Lagergren 准一级动力学模型, 同时其平衡吸附等温线能很好地符合 Langmuir 等温方程, 通过对吸附热力学研究发现, ΔG^\ominus 计算值为负, ΔH^\ominus 计算值为正, 这表明此类吸附本质上为吸热自发过程。吸附机理表明苯甲酸分子在层间是倾斜的, 与羟基层形成了一个角度。

关键词: Cu-Zn-Al-CO₃-LDH; 苯丙氨酸; 吸附; 机理

(Edited by LI Xiang-qun)