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Lignocellulose-Montmorillonite Nanocomposite as Adsorbents of Pb(II) in Aqueous Solution: The Capacity for Desorption and Regeneration

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Lignocellulose-montmorillonite nanocomposite was used as adsorbent for the removal of Pb(II) from aqueous solution. The effects of each desorption parameter were discussed, the optimal desorption capacity of Pb(II) was found to be 105.2 mg/g by using HNO_3 as regenerating agent, when the optimal initial HNO_3 concentration was 0.03 M, the optimal desorption temperature was 30 ºC, the optimal desorption time was 0.5 h. SEM, XRD and FTIR were used to characterized the type of functional groups for lignocellulose-montmorillonite and Pb(II) ions binding process. In addition, five adsorption/desorption cycles were tested and confirmed that lignocellulose-montmorillonite was suitable for regeneration, it indicated that lignocellulose-montmorillonite can be utilized as a low-cost sorbent for the removal of Pb(II) ions from wastewater.

Keywords: Desorption, Lignocellulose-montmorillonite, Nanocomposite, Pb(II), Regeneration.

INTRODUCTION

Nowadays water resources shortage and pollution have received worldwide attentions. One major environment pollution in wastewater is caused by heavy metals[1], such as mining, refining ores, tanneries, leather, plastics, paper, electroplating and painting industries. Unlike organic contaminants, most heavy metals in effluent water do not undergo microbial or chemical degradation, furthermore these effluents are remained, accumulated, migrated throughout food chains and may lead to critical ecological issues and health problems. Among heavy metals, lead is one of the most widely distributed, its toxicities can cause hypertension, nephritis, cramps, nausea, behavioral changes *etc.*[2]. Thus, removing heavy metals from wastewater is important for preserving the ecosystem and for economic reasons[3].

Several techniques have been developed and used for removal of heavy metal ions, including precipitation, ion exchange, filtration, flotation, solvent extraction, membrane processing and adsorption[4]. Among these methods, adsorption is considered as an effective method, because of its high efficiency, easy handling, low cost, regeneration of adsorbents[5] and metal recovery. Therefore, there is an urgent need for cost-effective and friendly adsorbents[6].

Lignocellulose (LNC) is one of the most abundant renewable resources, widely exists in plant seeds. Lignocellulose has higher specific surface areas, larger molecular mass and a three-dimensional structure, existing aromatic groups, phenolic hydroxyl groups, hydroxyl and other active groups, which could be used as the active adsorption sites of heavy metal ions[7]. There have been many reports about the application technology of lignocellulose, because of its polydispersity and amorphous structure make it hard to be widely used. Montmorillonite (MMT) has being considered as an alternative low-cost adsorbent. It is a kind of layered aluminosilicate, possesses larger cation exchange ability and can also be easily obtained[8]. Recently, biodegradable natural polymer/montmorillonite nanocomposites have been investgated[9]. However, adsorption and desorption of the nanocomposites on heavy metal ions were rarely reported. Due to economic considerations and adsorption performance, we prepared the lignocellulose-montmorillonite (LNC-MMT) nanocomposite as adsorbent of Pb(II). Its unique structural and functional properties may lower the concentration of Pb(II) in an aqueous solution[10].

The present study is the first attempt to investigate the desorption behaviours of LNC-MMT nanocomposite for the removal of Pb(II), adsorption experiments on Pb(II) of LNC-MMT nanocomposite reported in another article[11]. Desorption conductions of Pb(II)-loaded LNC-MMT were screened by varying the initial HNO_3 concentration, contact time and desorption temperature. The desorption mechanism between the surface complexation and ion exchange[12] was examined by

X-ray diffraction (XRD), Fourier transform infrared (FTIR) and scanning electron microscopy (SEM) analysis. The obtained results can offer a reference to choose LNC-MMT as the appropriate material for Pb(II) removal and its regeneration ability was evaluated through five adsorption/desorption cycles and showed a good recycling.

EXPERIMENTAL

Lignocellulose (SAM-100), purchased from Beijing Huaduo Biological science and technology limited company, China. The cationic exchange capacity of montmorillonite (Zhejiang Fenghong Clay chemical industry limited company, China.) is 100 mequiv/100 g. Montmorillonite was ground and sieved to 200 mesh size. Pb $(NO_3)_2$ (Tianjin Fengchuan Chemical Reagent Co., China) was used to prepared the adsorbate solution. HNO_3 is purchased from Tianjin Zhengxing Chemical Reagent Acid Factory, China. Other agents used were analytic grade without further purification. All solutions were prepared with distilled water.

X-ray diffraction analysis of the powered samples were performed using an X-ray power diffractometer with Cu anode (PAN alytical Co. X'pert PRO), running at 40 kV and 30 mA, scanning from 4º to 18º at 3º/min. Micrographs of the samples were taken by scanning electron microscopy (SEM) (JSM-5600LV, JEOL, Tokyo, Japan). Before observation of SEM, all samples were fixed on aluminum stubs and coated with gold. FTIR spectra of the LNC-MMT samples were characterized by using a Fourier transform infrared (FTIR) spectrophotometer (Thermo Nicolet NEXUS; Thermo Fisher Scientific, Waltham, MA, USA) in KBr pelletys.

Preparation of LNC-MMT nanocomposite: Lignocellulose (4 g) was added to a 20 wt. % sodium hydroxide solution (lignocellulose quality:NaOH volume = 1:30) in batches, after magnetic stirring about 0.5 h at room temperature, forming a homogeneous suspension. Montmorillonite (4 g) was swelled by 120 mL distilled water, then lignocellulose-NaOH suspension was slowly added to montmorillonite solution stirring at 500 rpm for 0.5 h, followed the mixture was heated to 60 ºC by stirring for 6 h, centrifugal separation (500 rpm) by a high-speed refrigerated centrifuge (H-2050R), the composite was filtered and washed with distilled water until adjusted to pH 7, then dried in an oven (DZF-6210) at 105 ºC for 5 h, ground and sieved to 200 mesh size, used for further adsorption experiments[13].

Adsorption experiments: The adsorption experiments were carried out for a single component by batch techniques to determine the extent of adsorption of LNC-MMT on Pb(II). The desired dose of LNC-MMT 0.1000g (BS210S) was added to 50 mL Pb $(NO_3)_2$ of a certain concentration in a series of 100 mL conical flasks. The suspension was stirred on a magnetic stirrer at a uniform speed of 120 rpm in a thermostated shaker (SHA-C) and adjusted pH with a certain amount of CH_3COONa-CH_3COOH buffer solution using a pH meter (PB-10). After adsorption equilibrium was reached, the mixture was rapidly separated by centrifugation at 4500 rpm for 10 min. Both the initial and the final concentrations of Pb(II) ions were analyzed by a ethylenediaminetetraacetic acid (EDTA) titrimetric method using 0.05 M EDTA solution as a standard solution[14] and 0.2 % xylenol orange solution as a indicator[15]. All the data presented were the average of three experimental runs. The absorbencies of Pb(II) solution were measured from the following equation[16]:

$$Q_e = (n_o - n_e) \times 207.2/m_1 \quad (1)$$

where Q_e (mg/g) is the amount of Pb(II) ions adsorbed at equilibrium. n_0 (mol) is the initial molar volume of Pb(II) ions in the adsorption solution. n_e (mol) is the final molar volume of Pb(II) ions in the adsorption solution at equilibrium. m_1 (g) is the dose of the adsorbent. In the method of calculating Q_e, no losses of Pb(II) ions to any other mechanism (volatilization, sorption to the glassware, degradation, *etc*.) were assumed.

Preliminary experimental conditions were optimized, the results showed the equilibrium adsorption amount of Pb(II) is approximate 140.94 mg/g in the given optimization conditions of the dosage of LNC-MMT 0.1000 g, adsorption time 1 h, adsorption temperature 30 ºC, optimum pH 6.0 and the initial concentration of Pb(II) ions 0.04 M. More detailed contents of adsorption experiments on LNC-MMT will be publised[11].

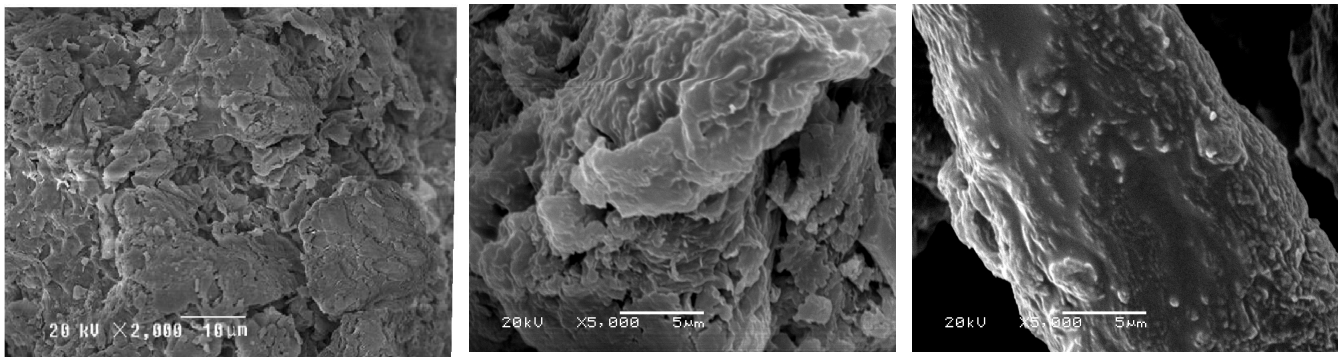
Desorption and regeneration of LNC-MMT: The batch desorption experiments were performed in an Ultrasonic cleaning machine (KS-300EI). To find effective conditions for desorption of the Pb(II) loaded LNC-MMT, a certain concentration of HNO_3 was used as the regenerating eluent.

50 mL HNO_3 solution was added to a series of 150 mL conical flasks, then 0.1000 g Pb(II)-loaded LNC-MMT was cautiously transferred into the conical flasks. The mixture was put in the ultrasonic cleaning machine. After the desorption equilibrium was reached, the suspension was centrifuged at 6000 rpm for 5 min, the pH was adjusted to the desired value by adding a certain amounts of CH_3COONa-CH_3COOH buffer solution and the concentration of Pb(II) ions in solution measured by a EDTA titrimetric method using 0.05 M EDTA solution as a standard solution and 0.2 % xylenol orange solution as a indicator. The influence of HNO_3 concentration on Pb(II) desorption was studied by agitating six different initial HNO_3 concentrations (range = 0-0.05 M) at 30 ºC for 0.5 h. The effect of time on Pb(II) desorption was performed for six predetermined time intervals (range = 5-120 min) by adding 0.03 M HNO_3 at 30 ºC. The effect of temperature on Pb(II) desorption was carried out at six different temperatures (range = 20-70 ºC) by adding 0.03 M HNO_3 until desorption equilibrium was established. For each condition, three experiments were performed and the reproducibility of the results was within ± 3 %. The desorption capacity of the Pb(II)-loaded LNC-MMT was calculated according to the following equation[17]:

$$Q_t = n \times 207.2/m_2 \quad (2)$$

where Q_t (mg/g) is Pb(II) concentration in acid solution at desorption equilibrium. n (mol) is the molar volume of Pb(II). m_2 (g) is the mass of the adsorbent after adsorption of Pb(II).

Repeated batch operations were performed to examine the reusability of LNC-MMT for Pb(II). Adsorption and desorption tests were carried out as above. The resulting composite was washed twice with distilled water to remove the remaining acid and was dried in an oven (DZF-6210) at 70 ºC for the next adsorption of Pb(II). The amounts of adsorption and desorption of Pb(II) were determined according to the aforementioned method. The consecutive adsorption/desorption process was performed five times.



(a) (b) (c)

Fig. 1. SEM images of LNC-MMT (a) and LNC-MMT after adsorption Pb(II) (b) (c)

RESULTS AND DISCUSSION

Morphology and structure of the LNC-MMT

SEM images analysis: To determined the surface morphology and elemental composition of LNC-MMT before and after adsorption Pb(II), SEM analysis has been carried out. The SEM images of raw LNC-MMT and Pb(II) -loaded LNC-MMT were shown in Fig. 1. It can be seen from Fig. 1 (a) that native LNC-MMT exhibited an irregular surface morphology, montmorillonite particles are uniformly distributed in the matrix structure of lignocellulose, it can be suggested that the disordered or exfoliated structure of LNC-MMT nanocomposite to be an appropriate surface area for metal ions adsorption[18,19]. The SEM images after Pb(II) adsorption were given in Fig.1 (b) and 1(c). The additional brighter spots were Pb(II) ions on the porous surface areas of LNC-MMT, the distributions of brighter spots were not evenly spread, indicating only selected functional groups were involved in the adsorption of Pb(II) ions, such as carboxyl and hydroxyl, owing to oxygen atoms can provide nonbonding electrons to coordinate with the divalent Pb(II). In other words, the carboxyl and hydroxyl on the LNC-MMT surfaces were considered as the dominant factors in the adsorption process and may be existed complexation potential mechanisms and ion exchanges[20].

X-ray diffraction analysis of the LNC-MMT: X-ray diffraction was an effective method for investigation of the intercalation existence of montmorillonite. Fig. 2 illustrated the XRD patterns of montmorillonite and LNC-MMT nanocomposite. The XRD pattern of purified montmorillonite (Fig. 2(a)) showed a typical diffraction peak at 5.83°, according to Bragg equation [$2d\sin\theta = k\lambda$ ($k = 1,2,3,\ldots$)], it was responding to a basal spacing of d = 1.48 nm. After intercalation with lignocellulose (Fig. 2 (b)), the movement of the typical diffraction peak of montmorillonite shifted to lower angle ($2\theta > 5.83°$), which indicated the formation of intercalated nanostructure. According to the results of XRD, it can be concluded that almost all lignocellulose intercalated into montmorillonite interlayer with destroying the crystalline structure of montmorillonite.

Infrared spectroscopy analysis: The FTIR spectroscopic studies were carried out to understand the surface functional groups that responsible for the interaction of LNC-MMT and Pb(II) ions. Fig. 3 showed the FTIR spectra of LNC-MMT before and after Pb(II) adsorption. The results indicated that

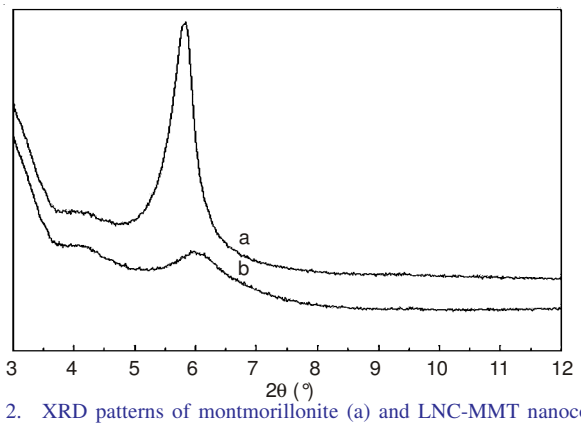


Fig. 2. XRD patterns of montmorillonite (a) and LNC-MMT nanocomposite (b)

the wide adsorption band at 3445 cm^{-1}, corresponding to the stretching vibration of O-H groups, become stronger and wider after adsorption Pb(II), due to part of formed hydrogen bonds broken away in the adsorption process and caused the stretching vibration of hydroxyl shifting relatively lower and wider peak position. The peak at 2922 cm^{-1} was asymmetric stretching vibration of methoxy connected with benzene ring of the lignocellulose. The adsorption band at 1637 cm^{-1} assigned to the O-H bending vibration of H_2O of the montmorillonite, strengthened and shifted to lower wave number 1569 cm^{-1}, which attributed to the -COOH groups asymmetric stretching vibration of intercalated lignocellulose (1600 cm^{-1}) overlapping with -OH bending vibration of H_2O of the montmorillonite. The peak observed at 1422 cm^{-1} was the symmetric stretching vibration of -COOH, shifted to lower wave number 1420 cm^{-1} and become obviously stronger, may be assigned to the ion exchange reactions between carboxyl anion and Pb(II). In addition, the adsorption band at 1027 cm^{-1} was Si-O stretching vibration, widen and shifted to lower wave number 1025 cm^{-1}, can be assigned to the ligand reaction and complexing reaction between oxygen atom of Si-O and Pb(II). The information from FTIR spectra concluded that the -OH and -COOH groups of LNC-MMT were involved and played an important role in the adsorption of Pb(II) process[21].

Influencing factors on Pb(II) desorption

Effect of HNO_3 concentration on Pb(II) desorption: The acidity of the solution was one of the most important factors influencing the adsorption and desorption behaviors of Pb(II) ions on LNC-MMT[22]. The effect of initial HNO_3 concentration

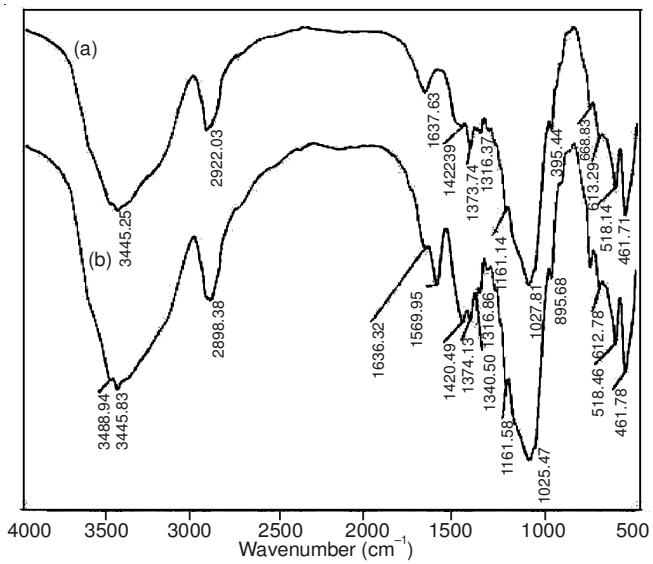


Fig. 3. IR spectra of LNC-MMT (a) and Pb(II)-loaded LNC-MMT (b)

on desorption capacity of Pb(II)-loaded LNC-MMT was shown in Fig. 4. It can be seen that the desorption capacity was negligible below 0.01M initial HNO_3 concentration and increased sharply from 63.1 mg/g to approximately 105.2 mg/g with increasing HNO_3 concentration from 0.01M to 0.03 M, then there was a little increase with further increasing HNO_3 concentration to 0.04 M. This result was attributed to the following facts, desorption of Pb(II)-loaded LNC-MMT below 0.01 M initial HNO_3 concentration, availability of negatively charged groups on LNC-MMT surface was occupied by Pb(II), so under lower initial HNO_3 concentration it was unlikely to withstand the attraction between LNC-MMT and Pb(II). In contrast, when initial HNO_3 concentration gradually increased, more H^+ and H_3O^+ existed in the solution systems, H^+ competed with Pb(II), resulting in more active sites to become deprotonated to the virtual exclusion of Pb(II) binding on LNC-MMT surfaces. Nevertheless, another plausible explanation for Pb(II) desorption can be provided that the presence of high concentration of H^+ ions in the solution, resulted in the electrostatic repulsion between both positively charged LNC-MMT surfaces and Pb(II) ions. Since lead hydroxide is amphoteric, it was increasingly soluble at lower acidic solution, thus during the process of desorption of Pb(II), relatively lower initial HNO_3 concentration (≤ 0.05 M) were maintained. When initial HNO_3 concentration secutively increased, the desorption equilibrium has already reached saturation and the desorption capacity remained unchanged. Based on these results, the initial HNO_3 concentration 0.03 M was chosen as the optimum concentration for further studies.

Effect of desorption time on Pb(II) desorption: It was essential to evaluate the effect of contact time required to reach desorption equilibrium for desorption experiments. The effect of desorption time on desorption capacity of Pb(II)-loaded LNC-MMT was shown in Fig. 5. It can be easily found that Pb(II) ions desorption increased sharply at short contact time until equilibrium reached. High desorption rate at initial stage may be explained as the availability of large number of H^+ ions competed with loaded-Pb(II) in the solution, then Pb(II) separated from the surfaces of LNC-MMT rapidly within 0.5 h

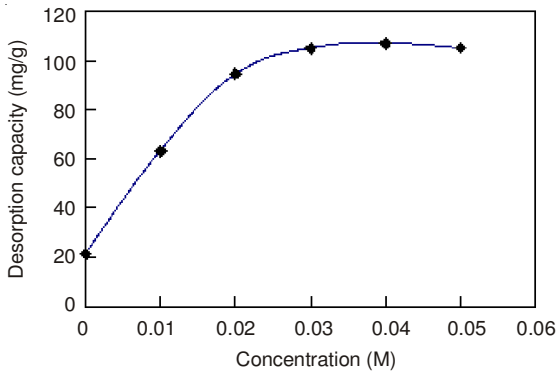


Fig. 4. Effect of concentration of HNO_3 on desorption capacity of Pb(II)-loaded LNC-MMT: for Pb(II) desorption experiment-sample dose, 0.1000 g; temperature, 30 ºC; equilibrium time, 0.5 h

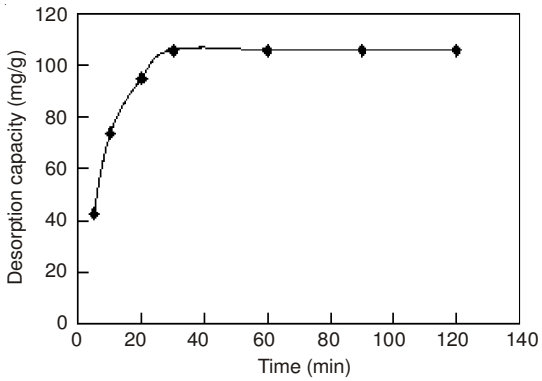


Fig. 5. Effect of desorption time on desorption capacity of Pb(II)-loaded LNC-MMT: for Pb(II) desorption experiment-sample dose, 0.1000 g; initial HNO_3 concentration, 0.03 M; temperature, 30 ºC

and advanced into acidic solution, releasing vacant surface active sites. In the later stage, desorption was likely slightly affected by the contact time. Then LNC-MMT renewed original framework, preparing for the next adsorption. The desorption equilibrium time was found to be 0.5 h, the highest desorption capacity was 105.2 mg/g.

Effect of desorption temperature on Pb(II) desorption: Generally the desorption capacity of Pb(II)-loaded LNC-MMT was dependent on the temperature[23]. The effect of temperature on desorption was studied at six different temperatures (range = 20-70 ºC) and the results were shown in Fig. 6. As shown in Fig. 6, the amount of desorption capacity increased with the desorption temperature. The amount of Pb(II)-loaded LNC-MMT desorption increased from 99.4 to 105.2 mg/g varying the temperature from 20 to 30 ºC, it indicated that the higher temperature was benefical to desorption, it could be due to the fact that increasing temperature may speed up the thermal motion of molecules and increase the collision probability of molecular between each other, at the same time higher temperature facilitated the physical desorption between Pb(II) and the surface on LNC-MMT[24]. When temperature was higher than 30 ºC, desorption equilibrium has been almost reached, temperature may be little affected the desorption balance. Therefore, the optimum desorption temperature was selected to 30 ºC in present study.

Recycling and reusability of LNC-MMT: Reusing of LNC-MMT keeps the processing cost down and opens the possibility of recovering Pb(II) extracted from the wastewater.

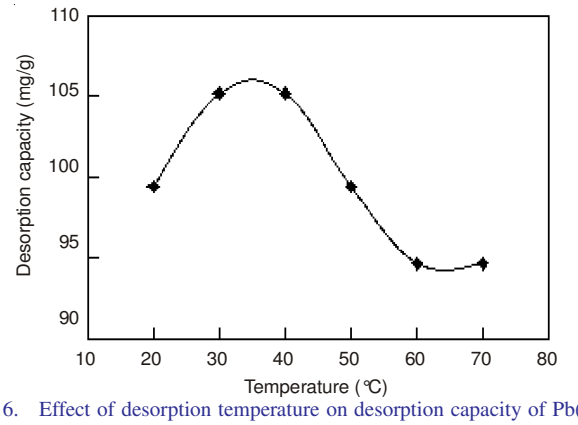


Fig. 6. Effect of desorption temperature on desorption capacity of Pb(II)-loaded LNC-MMT: for Pb(II) desorption experiment - sample dose, 0.1000 g; initial HNO_3 concentration, 0.03 M; equilibrium time, 0.5 h

Thus recycling of LNC-MMT was a very important parameter for its practical application. To evalutate the reusability of LNC-MMT, the consecutive adsorption/desorption processe was performed five times. The effect of reuse times on LNC-MMT adsorption/desorption capacity of Pb(II) was shown in Table-1. The recycled results indicated that the fading rate for Pb(II) solution was almost unaffected even after the forth run with regenerated LNC-MMT. This was attributed to the fact that a more stable network of LNC-MMT has been formed and the functional groups on the surfaces of LNC-MMT could interact with Pb(II) simultaneously. The synergistic effect of various functional groups was helpful to keep its adsorption capacity. In addition, LNC-MMT after desorption needed to be dried at 70 ºC, therefore each adsorption/desorption process must be disposed with HNO_3-treated process and a heated-treated process. This suggested that LNC-MMT nanocomposite was very suitable for the design to a continuous adsorption process and was a high-performance recyclable adsorbent.

TABLE-1
FIVE RECYCLES OF ADSORPTION AND DESORPTION OF LNC-MMT

Recycle times	1st	2nd	3rd	4th	5th
Adsorption Q_e (mg/g)	140.9	117.8	140.9	117.8	71.5
Desorption Q_e (mg/g)	105.2	94.7	94.7	94.6	48.4

Conclusion

In this work, novel LNC-MMT nanocomposite was synthesized by intercalation reaction of lignocellulose and montmorillonite. The optimal adsorption conditions of Pb(II) on LNC-MMT nanocomposite were the dosage of LNC-MMT 0.1000 g, adsorption time 1 h, adsorption temperature 30 ºC, optimum pH 6 and the initial concentration of Pb(II) ions 0.04 M, the adsorption capacity was 140.94 mg/g[11]. Desorption performance was strongly affected by parameters including initial HNO_3 concentration, desorption time and desorption temperature. Based on this research, the optimization desorption conditions were the dosage of Pb(II)-loaded LNC-MMT 0.1000 g, the optimum HNO_3 concentration 0.03 M, desorption time 0.5 h, desorption temperature 30 ºC, the highest desorption capacity obtained was 105.2 mg/g. FTIR and SEM analysis demonstrated that Pb(II) ions were adsorbed on selected surface active groups of LNC-MMT, due to non-specific cation exchange reactions (outer-sphere complexes)[25] and interactions with specific adsorption sites onto the adsorbent, where the adsorption mainly occurs in the micro-porous regions. Therefore both the ion exchanges and complexations may be the adsorption/desorption mechanisms of LNC-MMT relative to Pb(II). Moreover, LNC-MMT was found to be an effective adsorbent for the removal of Pb(II) ions from aqueous solution after five times of consecutive adsorption/desorption process. This work has built a solid foundation for the practical application of LNC-MMT as an adsorbent for the removal of Pb(II) and indicated that the LNC-MMT was a high performance, low-cost and recyclable green adsorbent for the wastewater treatment.

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