# 7th INTERNATIONAL CONGRESS ON ENVIRONMENTAL GEOTECHNICS

## LESSONS, LEARNINGS & CHALLENGES

10-14 NOVEMBER 2014 | MELBOURNE, AUSTRALIA

MELBOURNE CONVENTION AND EXHIBITION CENTRE

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ISBN 978-1-922107-23-7

Editors: Abdelmalek Bouazza, Sam Yuen, Bruce Brown

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# Pb(II) adsorption characteristics of loess modified soil-bentonite: equilibrium and non-equilibrium test

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## ABSTRACT

Equilibrium and non-equilibrium adsorption tests were carried out to investigate the characteristics of Pb(II) adsorption onto loess modified soil-bentonite (LSB). The effects of loess proportion in LSB, initial concentration of Pb(II), initial solution pH value, shaking time of soil-solute mixture were considered. The adsorption capacity of LSB increases linearly with the increasing of amount of loess and 20% of loess added to SB increases the adsorption amount by 2 times. Langmuir, Freundlich and D-R adsorption models were adopted to describe the experimental isotherms and isotherm constants. Equilibrium data fitted very well to the Langmuir model. The maximum monolayer adsorption capacities obtained from the Langmuir model are very large, which are 57.5 and 100 mg/g for SB and LSB respectively. The mean free energy of adsorption by D-R model is 15.56 and 18.89 kJ/mol for SB and LSB respectively, indicating the adsorption mechanisms of SB and LSB are mainly ion exchange and chemical reaction procedures. Pseudo-first and pseudo-second models were applied to describe the non-equilibrium experimental results and pseudo-second model fit better than pseudo-first model for both SB and LSB. The rate constant of second-order sorption of LSB is two times greater than that of SB, which indicates that the adsorption rate of LSB is two times faster than SB. Solution pH also has great effect on adsorption of Pb onto the soil samples. Higher pH values result in better adsorption capacity. Adsorption amount of LSB is 1.3-1.7 times greater than that of SB.

*Keywords:* cut-off wall, soil-bentonite, loess modification, lead, adsorption, batch-type test

## 1 INTRODUCTION

Soil-bentonite (SB) wall is widely used as vertical cutoff wall to prevent contamination of subsurface soil and groundwater. Conventional design of SB wall is predicated upon achieving a low backfill hydraulic conductivity, to inhibit groundwater flow and advective contaminant transport. However, low hydraulic conductivity may not be sufficient to ensure that an SB wall provide effective containment over a long time span (e.g., decades) and recently contaminant adsorption has been studied as a potentially significant mechanism for passively delaying the detrimental influence of diffusion through SB barriers (e.g., Krol and Rowe 2004; Malusis et al. 2010).Loess is widely distributed in several places throughout the world, such as Middle Asia, Russia, Middle East, North America and China (Tang et al. 2009). It is shown that loess has high adsorption capability for heavy metals such as Pb(II), Cu(II), Zn(II) and Cd(II) (Tang et al. 2008a,b, Li et al. 2009; Tang et al. 2009; Yang et al. 2012). There is a great potential to use loess to increase the heavy metal adsorption capacity of SB walls. The adsorption mechanisms of Pb(II) onto individual soils, such as bentonite and loess have been studied in the literature (Eren 2009; Wang et al. 2009; Hamidpour et al. 2010). Results show that the adsorption behaviour is affected by the concentration of solution, pH, soil/water ratio, temperature, and adsorption time etc. However, the adsorption mechanisms of mixed soils, i.e., silt-bentonite and loess modified soil bentonite have not been investigated in detail.

This paper aims to demonstrate the adsorption mechanism of silt-bentonite and loess modified soil bentonite. Both equilibrium and non equilibrium adsorption tests were carried out. The influence of the various experimental conditions including initial solution concentration, reaction time, and solution pH on the adsorption process were discussed.

## 2 MATERIALS AND METHODS

### 2.1 Materials

Three types of soil were selected, i.e., silt (S), loess (L) and Na-bentonite (B). Silt was sampled from shore side of Qiantang River, Hangzhou, China, which is typically a type of silty soil. Loess was sampled from Xi'an, which is a city located in the west of China. The geologic age of the loess is $Q_3$. Na-bentonite is a highly qualified natural Na-based bentonite generated from Wyoming State of the US. Silt and loess were dried at 105 $^oC$ for 24 hours, grounded into powder, sieved through a 2 mm mesh, and then put into plastic bags for storage. Na-bentonite was also dried at 105 $^oC$ for 24 hours and then stored in a two layered plastic-oil paper bag. Pb(II) solution (1000 mg/L) was prepared by dissolving lead chloride (analytical reagent grade) into deionized water (DW). The percentage of silt and bentonite (SB) are 5% and 95% by mass in silt-bentonite. The percentage of silt, loess and bentonite in loess modified silty bentonite (LSB) are 75%, 20% and 5% by mass, respectively.

### 2.2 Soil characteristics

Grain size distribution of silt and loess were determined. Particles larger than 0.075 mm were tested by a series of sieves and the remaining soil was tested using the sedimentary methods. The cation exchange capacity (CEC) of silt, loess and bentonite was determined by using the ammonium exchange method. The specific surface area was determined by $N_2$ adsorption method and the results were analyzed by Brunauer-Emmett-Teller (BET) adsorption theory. The natural pH ($pH_{na}$) of silt, loess and bentonite was determined by mixing 50 mg of soils with 25 mL of DW. The pH of solution was measured with a glass electrode potentiometer (multi 3240 SET B, Germany). To test the hydraulic conductivity of SB and LSB, the saturated SB and LSB slurry were prepared by a vacuum stirring machine and consolidated in organic glass tubes with pressure loads increased step by step. The highest pressure load here is 400 kPa. The hydraulic conductivity of SB and LSB columns under each pressure load was determined by the consolidation test.

### 2.3 Batch type tests

For non-equilibrium adsorption tests, 25 mg samples were mixed with 50 mL Pb(II) solution (the initial concentration is 200 mg/L) and then equilibrated at 25 $^oC$ on a shaking box at 180rpm. Samples were collected to determine the solute solution at 10 min, 20 min, 30 min, 1 h, 2 h, 4 h, 8 h, 12 h and 24 h after batch test started. For equilibrium adsorption batch test, 25 mg samples of soils were mixed with 50 mL Pb(II) solution with the concentrations ranging from 20, 50, 100, 200, 400, to 600 mg/L. The tests were also conducted at 25 $^oC$ on a shaking box at 180rpm for 24h. The initial pH values of the solution in non-equilibrium and equilibrium adsorption batch test were 8.5 ±0.5 and were not adjusted during the tests. To investigate the effect of solution pH on lead adsorption, 25 mg samples of the soils were mixed with 50 mL Pb(II) solution with the initial concentration 200 mg/L. The initial pH values of the solution were adjusted between 2 and 10 by adding 0.1 M HCl or NaOH to the solution. These tests were also conducted on the same box at 180 rpm for 24h at 25 $^oC$.

The supernatant of soil/solution at the end of the tests were obtained after centrifuged at 3000 rpm for 10 minutes. Pb(II) concentration and pH of the supernatant were determined by Atomic Absorption Spectroscopy (AAS) and a potentiometer, respectively. A duplicate of each type of tests and the control were conducted in this study.

### 2.4 Data analysis and modeling

The adsorption amount of soil samples towards Pb(II) is calculated by

$$S_{ad} = \frac{(C_i - C)V}{m} \tag{1}$$

where $S_{ad}$ is written as $S_e$ and $S_t$ in equilibrium and non-equilibrium test, respectively; $C_i$ (mg/L) is the initial concentration of Pb(II); C (mg/L) is the concentration of Pb(II) during the adsorption procedure and is written as $C_e$ (mg/L) and $C_t$ (mg/L) in equilibrium and non-equilibrium test, respectively; m (mg) is the mass of soil samples; and V (mL) is the volume of Pb(II) solution.

Two non-equilibrium adsorption models including pseudo-first order model and pseudo-second order model were adopted to evaluate the effect of contact time on the Pb(II) adsorption onto soils.

The pseudo-first order model is as follows (Do 1998):

$$\lg(S_e - S_t) = \lg S_e - \frac{k_1}{2.303}t \qquad (2)$$

where $S_t$ (mg $g^{-1}$) is the adsorption amount at time t; and $S_e$ (mg $g^{-1}$) is the equilibrium solute concentration; and $k_1$ ($\times 10^{-3}$g $mg^{-1}$ $min^{-1}$) is the rate constant of first-order sorption.

The pseudo-second-order model is as follows(Do 1998):

$$\frac{t}{S_t} = \frac{1}{k_2 S_e^2} + \frac{1}{S_e}t \qquad (3)$$

where $k_2$ (g $mg^{-1}$ $min^{-1}$) is the rate constant of second-order sorption.

Three isothermal equations, i.e., Langmuir, Freundich, and Dubinin- Radushkevich (D-R) models were adopted to evaluate the test results. The Langmuir model assumes a homogeneous monolayer surface adsorption and usually suits the whole equilibrium adsorption procedure (Sparks 2003):

$$\frac{C_e}{S_e} = \frac{1}{S_{max}K_L} + \frac{C_e}{S_{max}} \qquad (4)$$

where $S_{max}$ (mg/g) is the maximum adsorption capacity of the adsorbent; $K_L$ (L/mg) is the Langmuir constant.

The Freundlich model, which is based on sorption on a heterogeneous surface is given by (Sparks 2003):

$$S_e = K_F C_e^{1/n} \qquad (5)$$

where $K_F$ (L/g) and n are model constants, indicating the adsorption capacity and heterogeneity factor, respectively.
The D-R model is based on the concept of energy, which predicts the free adsorption energy change and explain the mechanism of adsorption (Do 1998):

$$\ln S_e = \ln S_{max} - k\varepsilon^2 \qquad (6)$$

where $S_m$ (mol/g) is the maximum adsorption capacity; k ($mol^2$/kJ) is a model constant related to the free adsorption energy and $\varepsilon$ is the Polanyi potential and can be written as

$$\varepsilon = RT\ln\left(1 + \frac{1}{C_e}\right) \qquad (7)$$

where R is gas constant (kJ $mol^{-1}K^{-1}$) and T is temperature (K).

The mean free energy of adsorption E (kJ/mol) is

$$E = -\frac{1}{\sqrt{2k}} \qquad (8)$$

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Basically, the mechanism of adsorption is physical adsorption, ion exchange, and chemical adsorption for |E| in the range of 1-8, 8-16 and larger than 16, respectively (Do 1998).

# 3 RESULTS AND DISCUSSION

## 3.1 Basic properties of soil samples

Table 1 shows the grain size distribution and correlated basic characteristics of silt, loess and Na-bentonite. The main grain component of silt and loess is silt particle, which are 80% and 66.7% by mass percentage, respectively. The silt contains more sand particles and less clay particles than loess do. Most of the soil particles of the bentonite are clay particle. The CEC of loess and bentonite are 6 times and 48 times than that of silt, respectively. BET surface area of loess and bentonite are 6 times and 19 times than that of silt, respectively. Natural pH of the silt, loess and the bentonite are 8.00, 8.05 and 8.53, respectively.

*Table 1: Grain size distribution of and correlated basic characteristics of soil samples*

| | Sand% 0.25-0.05mm | Silt% 0.05-0.005mm | Clay% <0.005mm | CEC meq/100g | Surface area m$^2$/g | $pH_{na}$ |
|---|---|---|---|---|---|---|
| **Silt** | 15 | 80 | 5 | 1.65 | 4.65 | 8.00 |
| **Loess** | 6.7 | 66.6 | 26.7 | 9.94 | 23.12 | 8.05 |
| **Bentonite** | 0 | 13.2 | 86.8 | 79.93 | 80.23 | 8.53 |

Table 2 shows the mineral components of the silt, loess and bentonite. The mineral components of silt and loess are the same, i.e., calcite, quartz, clinochlore, muscovite, albite and orthoclase, while the amount of each component differs. Bentonite contains different mineral component (quartz excepted) from silt and loess. The main mineral component of silt is quartz (54.6 %), which is 1.5 times of that of loess and 4.5 times than that of bentonite. Loess contains relatively large amount of calcite (14.8%) and bentonite contains large amount of Na-montmorillonite (71.4%).

*Table 2 : Mineral components of silt, loess, bentonite, and LSB*

| | Calcite | Quartz | Clinochlore | Muscovite | Albite | Orthoclase | Aldesine | Illite | Na-mont |
|---|---|---|---|---|---|---|---|---|---|
| **Silt** | 2.6 | 54.6 | 3.6 | 14.2 | 17.3 | 7.7 | 0 | 0 | 0 |
| **Loess** | 14.8 | 38 | 6.6 | 22.8 | 12.9 | 4.8 | 0 | 0 | 0 |
| **Bentonite** | 0 | 12.3 | 0 | 0 | 0 | 0 | 12.7 | 3.6 | 71.4 |

The hydraulic conductivity of SB and LSB columns under the pressure load of 400 kPa are $6.9\times10^{-10}$ m/s and $3.6\times10^{-10}$ m/s, respectively.

## 3.2 Non-equilibrium adsorption

Figure 1 shows the variation of adsorbed amount of Pb(II) onto the soil samples with time. SB was prepared with 5% bentonite by mass. LSB was prepared with 5% bentonite and 20% loess by mass. For loess and silt, the amounts of adsorbed Pb(II) increased continually with increasing reaction time until reaction reached the state of equilibrium. The time needed to achieve equilibrium state for loess and S is 2 and 12 hours, respectively. For bentonite, large amount (65 mg/g) of Pb(II) was adsorbed immediately after the chemical reaction started, and then the adsorbed amount of Pb(II) increased slowly with time. For SB and LSB, about 20 mg/g of Pb(II) was adsorbed just after the reaction started, and then the adsorption amount increase slowly and linearly with time.

The test data were fitted with the above mentioned kinetic models (see Figure 2). The obtained model parameters were shown in Table 3. It is indicated that the pseudo-second-order was more appropriate than pseudo-first-order since the calculated equilibrium adsorption amounts for all soil samples were closer to the experimental data and the correlated coefficients are larger than 0.99. The rate constant of second-order sorption of LSB is two times of SB, which indicates that the adsorption rate of LSB is two times faster than SB.

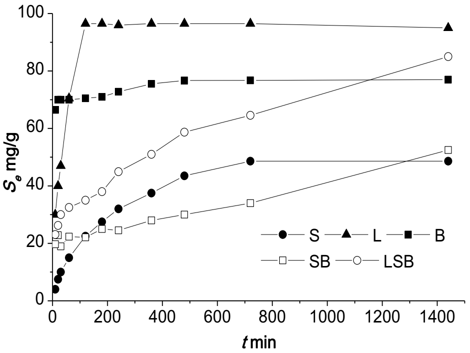
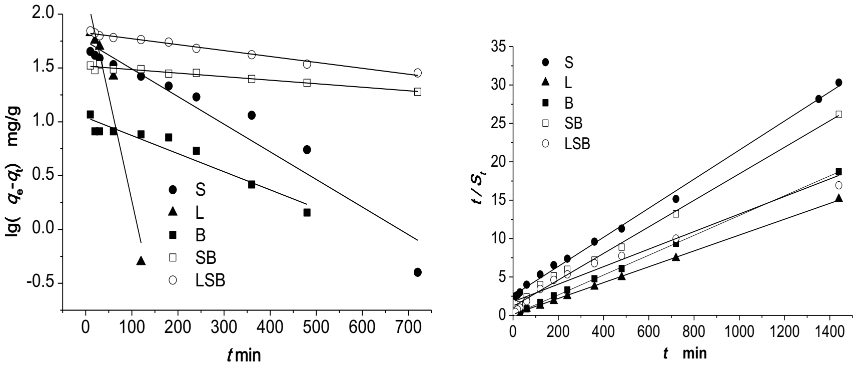


*Figure 1. Plots of Pb(II) adsorption kinetic test data (25°C, $C_i$=200 mg/L, s/w=2 g/L)*



(a) pseudo-first-order model (b) pseudo-second-order mod

*Figure 2. Linearized plots of non-equilibrium adsorption models*

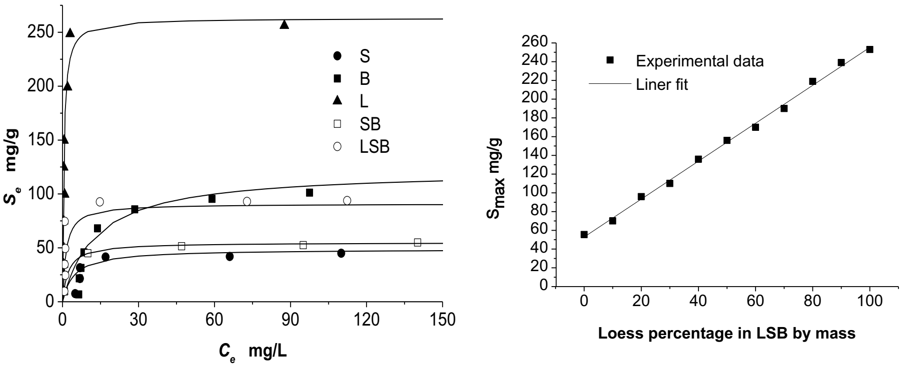
*Table 3: Predicted kinetic parameters of Pb(II) adsorption on silt, loess, bentonite, SB, LSB and LS*

| | Pseudo-first-kinetics | | | Pseudo-second-kinetics | | |
|---|---|---|---|---|---|---|
| | $S_e$ mg/g | $k_1$ $10^{-3}$min$^{-1}$ | $R^2$ | $S_e$ mg/g | $k_2$ $10^{-3}$gmg$^{-1}$min$^{-1}$ | $R^2$ |
| **S** | 56.00 | 5.988 | 0.935 | 52.91 | 0.138 | 0.996 |
| **L** | 83.18 | 18.654 | 0.992 | 97.09 | 0.771 | 0.999 |
| **B** | 60.02 | 3.224 | 0.910 | 77.52 | 1.949 | 0.999 |
| **SB** | 32.95 | 0.691 | 0.958 | 57.80 | 0.259 | 0.994 |
| **LSB** | 74.59 | 1.382 | 0.983 | 88.49 | 0.585 | 0.992 |

### 3.3 Equilibrium adsorption

The equilibrium adsorption data of Pb(II) onto silt, loess, bentonite, SB and LSB is shown in Figure 3(a). The shape of adsorption model for loess is a typical "H" type (Do 1998). The adsorption amount of Pb(II) per gram of soils ($S_e$) increased linearly as equilibrium Pb(II) concentration ($C_e$) increased from zero to an inflexion point, and then the $S_e$ increased slowly with $C_e$ until it reached the maximum adsorption capability of soils. The inflexion point of loess is the lowest among these 5 soils (i.e., 1.5 mg/L).This indicates that loess shows the greatest adsorption capability of Pb(II). This result agrees with that obtained by Li et al. (2009). The inflexion point of SB and LSB are 10.0 mg/L and 6.5 mg/L, respectively. This is due to the effect of the added loess on the silt-bentonite.

Figure 3(b) shows the adsorption amount ($S_{max}$) of LSB versus different percentage of loess in the LSB. The percentage of loess in these LSB samples rages from 0 to 100%. It is shown that the adsorption amount of lead on LSB increases linearly with the increase of loess percentage in LSB. 20% loess added to SB could double the maximum adsorption capability of SB. This indicated that loess plays a very important role in the increase of the adsorption amount of Pb(II) on the mixed soils.



(a) (b)

Figure 3. (a) Pb(II) adsorption isotherms on silt, bentonite, loess, SB and LSB; (b) Adsorption capability of LSB with different percentage of loess (25 °C, s/w=2 g/L, 24 h)

Table 3 shows the predicted model constants of test data with three adsorption models as shown in Eqs.(4)–(8). In general, the Langmuir model was found to be the best model to fit the adsorption data. The correlated coefficients of the Langmuir model ranges from 0.906 to 0.990. The maximum adsorption capacity of silt, loess, bentonite, SB, and LSB are 48.8, 263.2, 116.3, 57.5 and 100.0 mg/g respectively. Loess has a large adsorption capability for Pb(II), which is higher than a quarter of its quantity. This result agrees with that obtained by Li et al. (2009). It is shown that the adsorption amount of the LSB (with 20% loess) can be 2 times greater than the unamended SB. The parameter $K_F$ by Freundlich model of LSB is also two times greater than that of SB, which indicates LSB has higher adsorption ability than SB when the initial concentration of Pb(II) is the same. The free energy of adsorption $|E|$ by D-R model of loess and BLS is 22.36 and 18.89 kJ/mol, respectively. Both values of $|E|$ for loess and BLS are larger than 16. These results show that the adsorption mechanism of Pb(II) on these soil samples is mainly chemical reactions. The $|E|$ value of silt, bentonite and SB is 14.14, 12.31 and 15.56 kJ/mol, respectively, indicating that the main adsorption mechanism of Pb(II) onto silt , bentonite and SB is ion exchange.

*Table 3 Predicted isothermal constants by Langmuir, Freundlich and D-R isotherms*

| | Langmuir | | | Freundlich | | | D-R | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | $K_L$ L/mg | $S_{max}$ mg/g | $R^2$ | $K_F$ L/g | n | $R^2$ | $q_m$ mol/g | K $mol^2/kJ$ | /E/ $mol^2/kJ$ | $R^2$ |
| **S** | 0.094 | 48.8 | 0.956 | 32.03 | 14.7 | 0.93 | 0.000492 | 0.0025 | 14.14 | 0.591 |
| **L** | 2 | 263.2 | 0.97 | 173.4 | 8.88 | 0.66 | 0.002035 | 0.001 | 22.36 | 0.726 |
| **B** | 0.17 | 116.3 | 0.978 | 33.14 | 4.13 | 0.88 | 0.001725 | 0.0033 | 12.31 | 0.859 |
| **SB** | 0.104 | 57.5 | 0.96 | 39.86 | 16.69 | 0.95 | 0.000496 | 0.0016 | 15.56 | 0.502 |
| **LSB** | 0.185 | 100 | 0.99 | 77.24 | 15.77 | 0.91 | 0.000777 | 0.0014 | 18.89 | 0.923 |

### 3.4 Effect of pH

Figure 4 (a) and (b) show the effect of initial pH value ($pH_i$) of the solution on the equilibrium adsorption amount ($S_e$) of Pb(II) onto the soils (i.e., the loess, silt, bentonite, SB and LSB) and on the equilibrium pH value ($pH_e$) of the solution, respectively. As shown in Figure 4(a), for Pb(II) solution with initial concentration of 200 mg/L, loess adsorbed 100% of Pb(II) even at low pH values. The adsorption amount of silt, bentonite, SB and LSB towards Pb(II) increase as the increase of $pH_i$. The adsorption amount of LSB is 1.3 to 1.7 greater than that of SB at different pH values. This indicates loess has a high adsorption ability for Pb(II) even in acid environment and could increase the adsorption ability of SB in acid environment.

It is shown in Figure 4(b) that $pH_e$ increases with the increase of $pH_i$ for all soil samples, then remains at a certain value. The stable pH value of loess is 8, which is higher than that of other soil samples (about 6). Loess contains large amount of calcite (14.8%, chemical component is $CaCO_3$) and when pH<8, $CaCO_3$ is dissolved (Li et al. 2009) until pH value reaches 8.In silt and bentonite, orthoclase and andesine contains -OH (Mitchell and Soga 2005) and may play roles in the adjustment of pH value by the chemical reactions (Sari et al. 2007). Results also indicates that these soil samples have the ability to adjust the pH of the solutions and the ability of loess adjusting pH value is higher than other soil samples.

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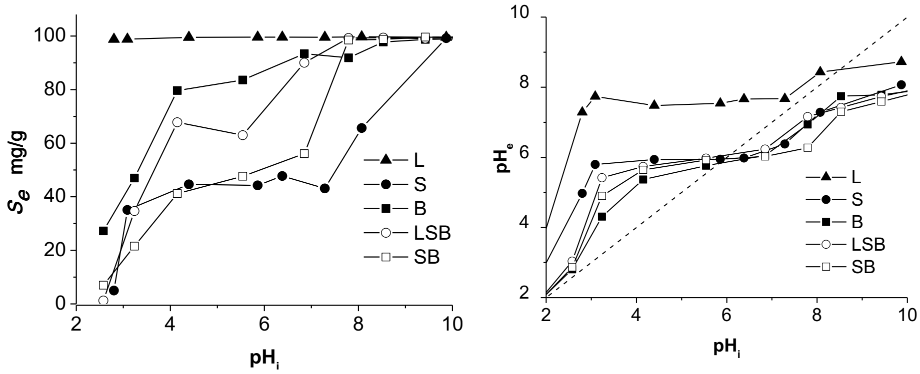

(a) (b)

Figure.4 Effect of initial pH on the equilibrium adsorption amount (a) and the equilibrium pH (b) of Pb(II) onto loess, silt, bentonite, SB and LSB

## 4 CONCLUSIONS

The Chinese loess was added to the traditional soil-bentonite to improve its adsorption capacity. Equilibrium and non-equilibrium batch type tests were carried out regarding the unamended soil-bentonite and the loess modified soil-bentonite. The conclusions are as follows:

(1) Equilibrium adsorption data of Pb(II) onto SB and LSB were well fitted with Langmuir isotherm. Adsorption capacity of LSB increases linearly with the increase of loess amount and the adsorption capability of loess amended LSB (loess percentage is 20% by mass) can be 2 times than that of the unamended SB. The hydraulic conductivity of LSB was increased by a factor of 1.9 with the added loess. However, the value of the hydraulic conductivity of LSB is in the order of magnitude of $10^{-10}$ m/s, which can meet the requirement of standard vertical wall (i.e., hydraulic conductivity<$10^{-9}$ m/s).

(2) By comparing the adsorption data with the D-R isotherm, it is indicated that the mechanism of adsorption of Pb(II) onto loess is chemical reactions and the adsorption mechanism for silt and bentonite is ion exchange. It is also indicated the adsorption mechanism of loess amended silt-bentonite is chemical reactions, indicating that the loess plays an important role in the adsorption of LSB towards Pb(II).

(3) Non-equilibrium data for Pb(II) adsorption onto SB and LSB were well fitted with Pseudo-second adsorption and the rate constant of second-order sorption of LSB is two times greater than that of SB, which indicates that the adsorption rate of LSB is two times faster than SB.

(4) pH value has great effect on the adsorption of Pb(II) onto soils. The adsorption amount of LSB towards Pb(II) (initial concentration is 200 mg/L) is 1.3 -1.7 times greater than that of SB at different pH values. The soil samples have the ability to adjust the pH of the solutions and the ability of pH adjusting of loess is higher than the other soil samples.

## 5 ACKNOLEDGEMENTS

The authors would like to express the sincere gratitude to National Natural Science Foundation of China (Grant Nos. 51278452, 51008274 and 51108293), the Fundamental Research Funds for the Central Universities (Grant No.2014QNA4019), the National Basic Research Program of China (973 program) (Grant No.2012CB719806), Zhejiang Provincial Governmental public industry research special funds for projects (Grant Nos.2011C21061 and 2014C33015) and Zhejiang Provincial Natural Science Foundation (Grant No. LY13D060003) for the financial support to this study.

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