

# Role of Organic Acids in Desorption of Mercury from Contaminated Soils in Eastern Shandong Province, China

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**Abstract:** Batch experiments were conducted to study the effects of low-molecular-weight organic acids (LMWOAs) on desorption of Hg from two kinds of soils in the eastern Shandong Province, China. Of all LMWOAs, oxalic and citric acids were chosen as the representatives in this experiment because they are most common in soils. Desorption of Hg increases with the increase of extracting time, until the highest desorption amount at 6–8 h, and then declines. It indicates that timeliness is very important when organic acids are used for phytoremediation in contaminated soils. When the concentrations of organic acids increase from 1.0 mmol/L to 10.0 mmol/L, Hg desorption increases significantly regardless of oxalic or citric acid. Compared with oxalic acid, citric acid is more effective to enhance the desorption of Hg, especially from fluvo-aquic soil. With the increase of initial pH value from 3 to 8, Hg desorption decreases directly in the presence of oxalic acid. However, Hg desorption first decreases, then increases, and eventually decreases again in the presence of citric acid due to the variation of competitive ability between chelating of citric acid and adsorption of soil in different pH values. Citric acid showed greater ability to release Hg from soils than oxalic acid. According to the results of the present study, it is found that the bioavailabilities of heavy metal can be improved with selecting suitable types and concentrations of organic acid amendment and reasonable soil condition.

**Keywords:** mercury (Hg); organic acids; desorption; heavy metal; kinetics

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## 1 Introduction

Mercury (Hg), one of the most toxic heavy metals, has been recognized as a global environmental contaminant because of its volatility and toxicity (Nriagu and Christian, 2004; Zahir *et al.*, 2005; Wang Z W *et al.*, 2007). In China, metal smelting and coal combustion are the two major sources of Hg contamination in the environment (Zhang and Wong, 2007). Hg in soil can be absorbed by crops and accumulated in human body through food chains. Hence, reducing Hg uptake by roots is one of the focuses of environmental science.

Sorption and desorption are the most important che-

mical processes affecting the bioavailability of metals in soils (Lockwood and Chen, 1973; Schuater, 1991). Many factors (Hg speciation, soil pH, organic matter, soil colloids, inorganic ions, *etc.*) affect Hg adsorption-desorption process. Among these factors, organic acids are important parameters affecting the ability of soil to retain heavy metals such as Hg. Low-molecular-weight organic acids (LMWOAs) are natural products of root exudates, microbial secretions, and plant and animal residue decomposition in soils (Strom, 1997; Zhang *et al.*, 1997; Cristofaro *et al.*, 1998). Functional groups in LMWOAs can influence solubility and uptake of ions in soils through acidification, chelation, precipitation and

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oxidation-reduction reactions. The positions and types of the functional groups are of importance in determining the properties of metals complexing with organic acid and transport ability of metals (Burchhard *et al.*, 1995). Different organic acids may play various roles in affecting interaction between metal and soil. It is therefore important to understand the role of these organic acids in affecting the behavior of Hg in soils.

There has been much research on sorption and desorption of heavy metals by soils in the presence of organic acids (Nigam *et al.*, 2001; Bäckström *et al.*, 2003; Yang *et al.*, 2006; Yuan *et al.*, 2007; Pérez-Novo *et al.*, 2008). However, few studies have been done on Hg desorption behavior influenced by organic acids. In limited literatures, Yang *et al.* (2008) studied the effects of dissolved organic matter on Hg adsorption and desorption in soil. Although Jing *et al.* (2007) analyzed the Hg desorption from soils in the presence of organic acids, the desorption process was carried out after soil adsorption experiments. Little work has been done on the effect of organic acids on Hg desorption from contaminated soils.

In this paper, we selected citric and oxalic acids, existing commonly in soils, to investigate desorption characteristics of Hg from contaminated soils in the presence of organic acids. It is essential to understand the impact of different organic acids on the environmental behavior of Hg in the soil ecosystem and subsequent food chain contamination.

## 2 Materials and Methods

### 2.1 Soil sampling and analyses

The two soil samples used in this study were brown soil and fluvo-aquic soil taken from Zhaoyuan City and Laiyang City, respectively. They are important agricultural soils in the eastern Shandong Province, China.

Artificial contaminated soils were used in this study. Two pots containing 600 g brown soil or fluvo-aquic soil were added with HgCl<sub>2</sub> solution. Soils in pots were equilibrated for one month in saturated-paste water con-

dition. The subsamples of cultured soils (about 200 g) were air dried, ground and sieved through 0.25 mm mesh for desorption experiments and 0.149 mm for total Hg analysis. Others were sieved through 1 mm mesh for analysis of physical and chemical properties. Soil physical and chemical properties were measured by using the methods recommended by Lu (2000). Soil pH was measured in a 1 : 2.5 soil/water suspension with a combination electrode. Organic matter (OM) was determined with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> oxidation at 180°C. Cation exchange capacity (CEC) was determined by using the method of displacing exchangeable cations on soil particles with NH<sub>4</sub><sup>+</sup> followed by NH<sub>4</sub><sup>+</sup> determination. Particle-size distribution was measured by fractionation using wet sieving and sedimentation. Total nitrogen (N) was determined by Kjeldahl digestion and distillation. Available phosphorus (P) was extracted with 0.5 M NaHCO<sub>3</sub> by the Olsen method. Available Fe was extracted with diethylene triamine pentacetate acid (DTPA) and determined by atomic absorption spectrometry (TAS-990F, Persee, Beijing). With reference to the methods of Liu *et al.* (2003), total Hg was determined by H<sub>2</sub>SO<sub>4</sub>-HNO<sub>3</sub>-V<sub>2</sub>O<sub>5</sub> digestion method and measured by cold vapor atomic absorption spectrometry (CVAAS) (F732-V, Huaguang, Shanghai). The soil properties are listed in Table 1.

### 2.2 Kinetics of Hg desorption

Batch method was used to study the desorption characteristics. Soil (1.00 g each) was weighed into 50-ml plastic centrifuge tubes. Each tube was added with 20-ml solution containing citric or oxalic acid. The pH value of 1 : 20 soil/water suspension was adjusted to 5.5 with 0.1 mol/L HCl or NaOH. It was shaken at 150 rpm in a constant 25°C for 1 h, 2 h, 4 h, 6 h, 8 h and 16 h in a vapourbathing constant temperature vibrator, then centrifuged at 5000 rpm for 10 min. The Hg concentration in the supernatant was determined by CVAAS. The kinetics experiment was carried out at two concentrations of organic acid (1.0 mmol/L and 5.0 mmol/L).

Table 1 Physical and chemical characteristics of soil

| Soil             | pH  | OM<br>(g/kg) | CEC<br>(cmol/kg) | Particle size (%) |               |           | Total Hg<br>(mg/kg) | Total N<br>(g/kg) | Available P<br>(mg/kg) | Available Fe<br>(mg/kg) |
|------------------|-----|--------------|------------------|-------------------|---------------|-----------|---------------------|-------------------|------------------------|-------------------------|
|                  |     |              |                  | 1–0.02 mm         | 0.02–0.002 mm | <0.002 mm |                     |                   |                        |                         |
| Brown soil       | 6.7 | 13.3         | 11.2             | 74.7              | 6.3           | 19.0      | 85.5                | 0.74              | 17.2                   | 12.5                    |
| Fluvo-aquic soil | 6.9 | 13.7         | 8.8              | 69.9              | 17.6          | 12.5      | 112.1               | 0.81              | 20.6                   | 17.8                    |

### 2.3 Desorption of Hg influenced by different types and concentrations of organic acids

Several plastic tubes containing 1.00 g soil were added oxalic acid or citric acid solution with the concentrations of 0 mmol/L, 1.0 mmol/L, 2.5 mmol/L, 5.0 mmol/L, 7.5 mmol/L and 10.0 mmol/L (pH 5.5). After shaking for 6 h at 150 rpm at 25°C, the supernatant was centrifuged at 5000 rpm for 10 min, then determined by CVAAS.

### 2.4 Desorption of Hg influenced by organic acids with various initial pH

Solution (soil/solution 1 : 20) with 5.0 mmol/L oxalic or citric acid was added to each tube. The initial pH of desorption solution were adjusted from 3 to 8 with HCl or NaOH. The suspension was shaken at 150 rpm in a constant 25°C for 6 h, and centrifuged at 5000 rpm for 10 min. Hg concentration in supernatant was measured as described above.

### 2.5 Quality control

All vessels used in this study were soaked in 15% HNO<sub>3</sub> (V/V) overnight prior to use and rinsed with deionized water. All reagents used were of analytical grade or better. Blanks were analyzed at the beginning, middle, and end of sample batch to ensure no background was above the method detection limit. Certified standard soil samples (GBW07401 (GSS-1)) were used to ensure precision in determining Hg concentration of soil. The recoveries were 89.7%–102.4% for five measurements. The minimum detection limit was 3.3 ng/g for soil and 0.5 µg/L for solution.

### 2.6 Statistical analysis

All experiments were replicated three times and mean values were presented. The data obtained were processed by Microsoft Excel 2003, and statistical analyses were conducted by statistical package SPSS 12.0.

## 3 Results and Analyses

### 3.1 Kinetics of Hg desorption

When no organic acids were added, little Hg was released from the two soil samples. The desorbed amount was 0.31 mg/kg and 0.19 mg/kg for fluvo-aquic soil and brown soil, respectively. Adding organic acids greatly enhanced the desorption of Hg from soil. The desorption amount of Hg from two soils in different extraction time

in the presence of organic acids was shown in Fig. 1 and Fig. 2. Hg desorption increased initially, then decreased with the extraction time. When the concentration of organic acids was low (1.0 mmol/L), Hg desorption influenced by citric acid reached the maximum at 8 h, with 7.6 mg/kg for brown soil and 7.1 mg/kg for fluvo-aquic soil, respectively. In contrast, in the presence of oxalic acid, the time reaching maximal desorbed amount was different for the two soils. It was 2 h for brown soil and 8 h for fluvo-aquic soil, respectively (Fig. 1). In the organic acids solution with high concentration (5.0 mmol/L), although the amount of desorbed Hg was different from that of low concentration, the time reaching maximal desorption was similar (Fig. 2). It indicates that the time achieving maximal desorbed amount of Hg is related to the type of organic acid and soil.

Compared with the change produced by low concentration of organic acids, the variation of Hg desorption with time produced by high concentration was greater. For example, in fluvo-aquic soil, desorbed amount increased with extraction time, and at 4–6 h it reached the maximum, thereafter it decreased rapidly. The desorbed amount of Hg at 16 h declined to 5.5 mg/kg for oxalic acid and 16.5 mg/kg for citric acid respectively, which was lower than the desorbed amount at 1 h (Fig. 2). The kinetics curves of Hg desorption show that the higher desorbed amount almost occurred at 6–8 h. But no significant variation was observed between the maximum of desorbed amount and the desorbed amount at 6 h ( $P > 0.05$ ). Accordingly, to insure the most desorbed amount, we selected 6 h as the uniform extraction time in the experiments to analyze the influence of concentrations of organic acids and initial pH on Hg desorption. The results of kinetic experiment showed that long reaction time may inhibit the Hg desorption from soils. It can be attributed to the decomposition of organic acids induced by microbes in a long reactive process (Singh *et al.*, 1996; Tao *et al.*, 2005). Moreover, soil buffer action may also reduce the amount of organic acid, and therefore decrease the release of mercury.

Citric acid has greater ability than oxalic acid in enhancing Hg desorption from soils. For 5.0 mmol/L concentration of organic acids, the maximal desorption amount improved more by citric acid than that by oxalic acid, which was 51.1% in brown soil and 56.8% in fluvo-aquic soil, respectively. It implies that citric acid could increase the dissolved Hg and accordingly im-

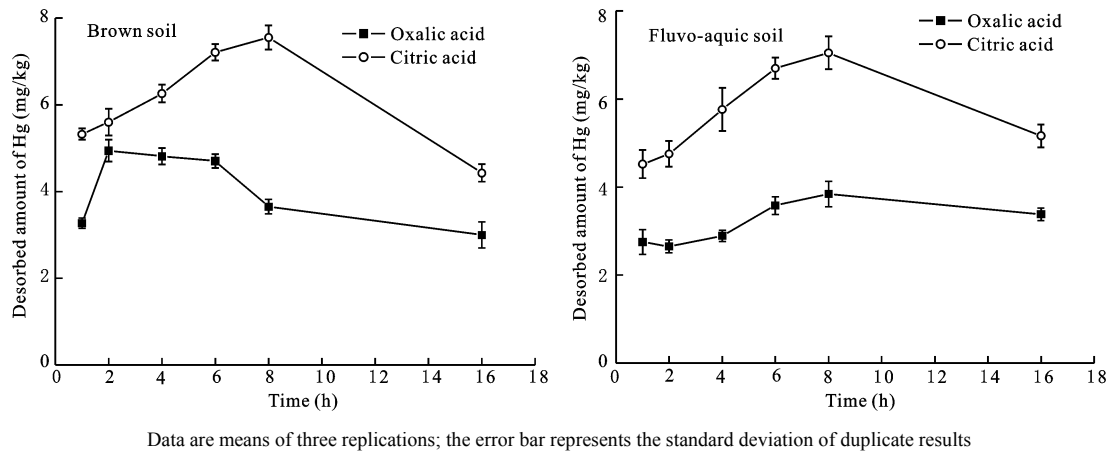


Fig. 1 Kinetics of Hg desorption from two soils influenced by organic acids with concentration of 1.0 mmol/L

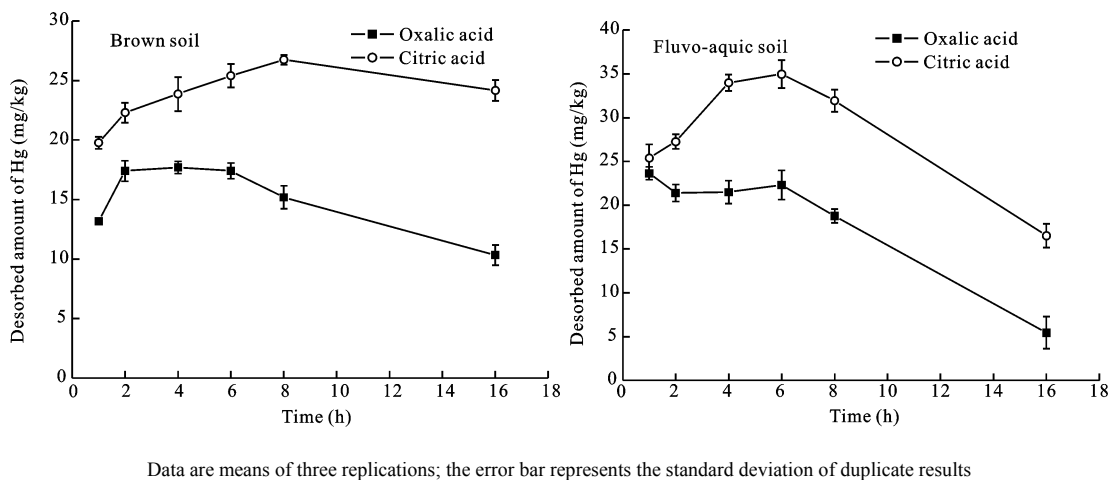


Fig. 2 Kinetics of Hg desorption from two soils influenced by organic acids with concentration of 5.0 mmol/L

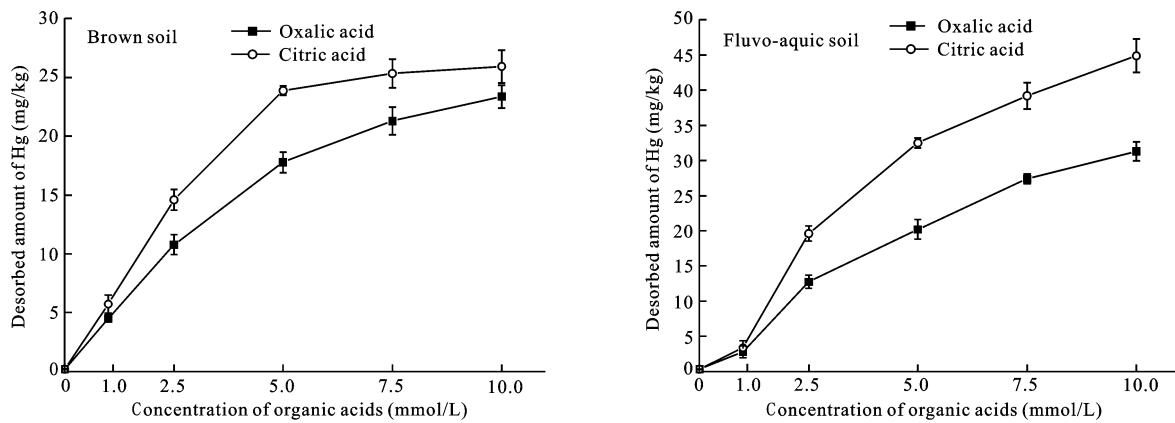
prove the bioavailability of Hg in soils. Therefore, citric acid has been used to enhance the accumulation of heavy metals by selected plants to amend the contaminated soils (Wu *et al.*, 2006; Duquène *et al.*, 2009). However, according to the result of kinetic experiment, it should be noticed that the timeliness is very important when the organic acids were added to soil for improving the efficiency of phytoremediation.

### 3.2 Influence of type and concentration of organic acids on Hg desorption

The batch desorption behavior of Hg was similar for the two soils (Fig. 3). The desorbed amount of Hg from the two soils increased with the increase of oxalic or citric acid concentration. The desorbed amount of Hg without organic acid was 0.19 mg/kg for brown soil and 0.31 mg/kg for fluvo-aquic soil. When citric acid concentra-

tion in the desorption solution increased from 1.0 mmol/L to 10.0 mmol/L, the desorbed amount of Hg increased from 5.7 mg/kg to 25.9 mg/kg for brown soil, and from 3.4 mg/kg to 44.9 mg/kg for fluvo-aquic soil. Similarly, when oxalic acid concentration in the desorption solution increased from 1.0 mmol/L to 10.0 mmol/L, desorbed amount of Hg increased from 4.5 mg/kg to 23.4 mg/kg for brown soil, and from 2.8 mg/kg to 31.3 mg/kg for fluvo-aquic soil respectively (Fig. 3). Table 2 shows the desorption ratio of Hg from the two soils. No significant variation of Hg desorption ratio was observed between two soil samples in the presence of oxalic acid ( $P > 0.05$ ), in spite of the greater desorption amount in fluvo-aquic soil. While in the presence of citric acid, the desorption ratio of Hg from fluvo-aquic soil was higher.

Yang *et al.* (2006) and Jing *et al.* (2007) found an inhibition to Hg or lead desorption from soil in the pres-



Data are means of three replications; the error bar represents the standard deviation of duplicate results

Fig. 3 Desorption curve of Hg under different concentrations of organic acids

Table 2 Desorption ratio of Hg from two soils under various concentrations of organic acids

| Concentration of organic acid (mmol/L) | Brown soil (%) |             | Fluvo-aquic soil (%) |             |
|--|----------------|-------------|----------------------|-------------|
|  | Oxalic acid    | Citric acid | Oxalic acid          | Citric acid |
| 0                                      | 0.2            | 0.2         | 0.3                  | 0.3         |
| 1.0                                    | 5.3            | 6.7         | 2.5                  | 3.0         |
| 2.5                                    | 12.6           | 17.1        | 11.4                 | 17.5        |
| 5.0                                    | 20.8           | 27.9        | 18.0                 | 29.0        |
| 7.5                                    | 24.9           | 29.6        | 24.4                 | 35.0        |
| 10.0                                   | 27.3           | 30.3        | 27.9                 | 40.1        |

ence of lower organic acid concentration ( $< 0.1$  mmol/L). It can be explained by the fact that small amount of organic acids adsorbed by soil components may increase negative charge or CEC of soil, thus desorbed Hg could be bound to the organic ligands which was adsorbed on the surfaces of the soil. However, In this study, the lower organic acid concentration did not show an inhibition to Hg desorption because the initial concentration (1 mmol/L) in the present study is far higher than that in previous studies. When the organic acid concentrations in desorption solutions increased, the ratio of organic ligands remained in solution to the organic ligands adsorbed by the soil rapidly increased, and accordingly the competitive ability of the organic ligands for adsorbing sites with Hg increased, thus enhancing desorption amounts with the increasing organic acid concentrations. The net desorption amount of Hg in the presence of organic acids is decided by the relative binding strengths between the soil surface sites and the metal, and the complex reactions between the organic acid and the metal (Wu *et al.*, 2003).

Through the formation of soluble complexes, organic

acids are able to impact the release of heavy metal from soil (Krishnamurti *et al.*, 1997). The organic ligands are not only adsorbed on the external surface, but also enter the interlayers of clay minerals. All of these factors influence the adsorption of Hg in soils. When the organic acids was added, it can chelate with  $\text{Hg}^{2+}$  or  $\text{HgOH}^+$ , thus resulted in the reduction of amount of adsorbed Hg onto soil surface. The stronger the chelator, the less  $\text{Hg}^{2+}$  adsorbed onto soil surface, and accordingly more Hg was desorbed. In this study, it was found that citric acid is stronger than oxalic acid in Hg desorption from soils.

The ability of organic acid in desorption process of Hg may depend on the molecular weight and structure of acid. In this study, citric acid has a higher molecular weight, thus can chelate more metals than oxalic acid, because it carries more negative charge and more surface area (Jing *et al.*, 2007). Moreover, according to the result of Jones and Barssington (1998), oxalic acid has a higher sorption in soil than citric acid. Adsorbed oxalic acid may increase the negative charge of soil surface, leading to a steady absorption, thus decrease Hg desorption from soils. In other aspects, the functional groups of

acids such as carboxylic and hydroxyl groups are important binding sites due to their higher complex ability. Citric acid has more functional groups such as carboxyl than oxalic acid, and accordingly it desorbs more  $\text{Hg}^{2+}$ .

### 3.3 Influence of initial pH on Hg desorption in presence of organic acids

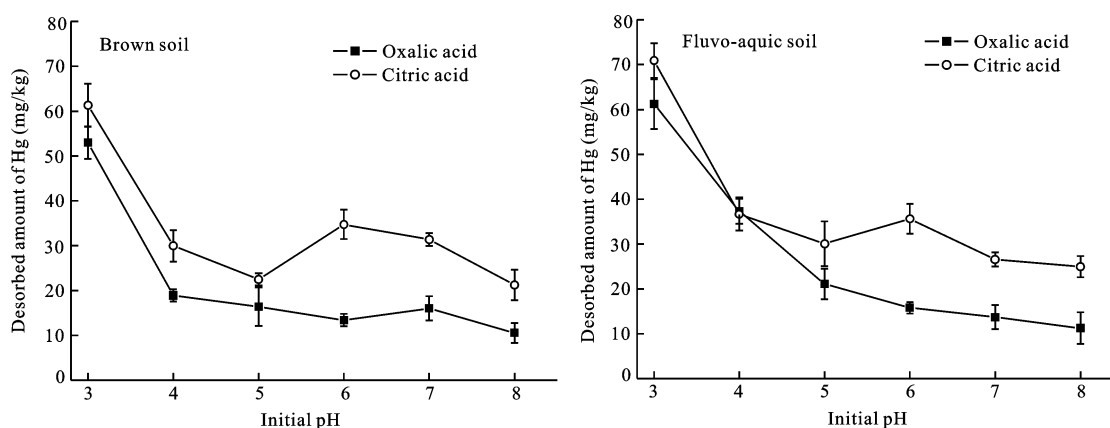
The change of Hg desorption from two soil samples under oxalic acid and citric acid solutions with varied initial pH is shown in Fig. 4. For both soil types, the desorbed amount of Hg decreased with the increase of initial pH from 3 to 8 in the presence of 5.0 mmol/L oxalic acid. The desorption curve can be divided into two stages. The rapidly decreasing stage was at pH 3–4 in brown soil, and at pH 3–5 in fluvo-aquic soil. At this stage, desorbed amount of Hg decreased sharply in the presence of oxalic acid from 52.9 mg/kg to 18.9 mg/kg for brown soil, and 61.2 mg/kg to 21.1 mg/kg for fluvo-aquic soil. The other pH range was slowly decreasing stage, and at this stage, Hg desorption showed a steady decline in the two soil types. Compared with oxalic acid, the desorption amount of Hg in the two soil types with varied initial pH in the presence of 5.0 mmol/L citric acid was not the same. It first decreased, then increased, and then decreased again with the increment of initial pH. The similar result was also found in previous study on other heavy metals (Gao *et al.*, 2003). It may be attributed to the property of citric acid.

According to the results of previous studies, the sorption amount of heavy metals in soils increased with the increase of pH (Lockwood and Chen, 1973; Yujun, 1996; Zhang and Song, 2005). As the opposite process of ad-

sorption, the desorbed amount of heavy metals should decrease with the increase of pH due to improvement of variable negative charges on the soil surface. However, when organic acids were added to soil solutions, it may change the desorption process of metals from soil, through the property of themselves. In the present study, oxalic acid showed little influence on Hg desorption with the increase of pH from 3 to 8, owing to its weak chelating ability with Hg. But the form of citric acid would change from  $\text{H}_3\text{L}$ ,  $\text{H}_2\text{L}^-$  and  $\text{HL}^{2-}$  to  $\text{L}^{3-}$  with the increase of initial pH, thus resulting in the variation of the chelating ability (Yin, 1985).  $\text{HL}^{2-}$  and  $\text{L}^{3-}$  rather than  $\text{H}_3\text{L}$  and  $\text{H}_2\text{L}^-$  were considered as better chelator to bind with heavy metals, which may lead to great desorption amount of Hg under the enhanced pH condition (Gao *et al.*, 2003; Wang Daichang *et al.*, 2007). In addition, in solutions with somewhat higher pH,  $\text{Hg}^{2+}$  may hydrolyze and exist as  $\text{HgOH}^+$ , which tended to be absorbed by soil, resulting in the decrease of Hg desorption. Therefore, the effect of citric acid on desorption behavior of Hg in various pH conditions is a multiple result of different influencing mechanism.

## 4 Conclusions

This paper investigated the desorption behavior of Hg from soils influenced by organic acids. The kinetics process and effects of initial pH and type of organic acids on the desorption of Hg was studied. The desorption mechanism in the presence of organic acids was also discussed. The results show that low-molecular-weight organic acids effectively improve the Hg desorption



Data are means of three replications; the error bar represents the standard deviation of duplicate results

Fig. 4 Effects of initial pH on Hg desorption from soils in presence of 5.0 mmol/L organic acids

from soils. The desorbed amount of Hg increases with the increase of extracting time, until the highest desorption amount occurs, and then declines. The desorbed amount of Hg enhances greatly when the concentrations of organic acids increase from 1 mmol/L to 10 mmol/L, regardless of the type of organic acid and soil. With the increase of initial pH from 3 to 8, the desorbed amount of Hg decreases under oxalic acid condition, while under citric acid condition, Hg desorption first decreases, then increases, and decreases again. Citric acid shows a greater ability to release Hg from soils than oxalic acid. The results of this study indicate that organic acid can be used to improve the efficiency of phytoremediation since it can enhance the bioavailability of Hg by promoting desorption of Hg in the soil. However, the amendment of organic acids is also influenced by the types and concentrations of organic ligands and pH value of soil solution.

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