Influence of Dissolved Organic Carbon on the Adsorption and Desorption of Cadmium in Reclaimed Soil

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ABSTRACT

Organic acids are widespread in the environment and play an important role in the adsorption, desorption, and migration of soil cadmium (Cd). Under the effect of humic acid (HA) and citric acid, the properties and mechanism of Cd adsorption and desorption in reclaimed soil from the Panyi Mine in the Huainan mining area were investigated (CA). The addition of HA and CA inhibited the adsorption and enhanced the desorption of Cd in reclaimed soil, with HA having a stronger effect than CA. As the concentration of added HA or CA increased, the adsorption capacity of Cd in the reclaimed soil gradually decreased, while the desorption capacity gradually increased. That is, the adsorption inhibition/desorption promotion effect was stronger for higher concentrations of organic acid. Cd adsorption and desorption could be described as a logarithmic function of organic acid concentration. The kinetic curves of Cd adsorption and desorption in reclaimed soil under the influence of organic acids show that both adsorption and desorption involved two stages: a fast reaction stage followed by a slow reaction stage..

INTRODUCTION

Coal mining has been going on in the Huainan mining area for over a century. Significant-scale coal mining has resulted in large amounts of solid waste and coal gangue, as well as land subsidence, with the subsidence area expanding. Since 2003, in the Huainan mining area, coal gangue filling and soil covering techniques have been employed to backfill subsided terrain with coal gangue to the original surface height, allowing the area to be reused (Bian 2001). However, reclamation activities have affected the soil quality in the mining area to a certain extent. The soil is easily compacted, has a low nutrient content and large volume, and is potentially contaminated by heavy metals. In particular, the content of the heavy metal cadmium (Cd) has exceeded the agricultural land filter value for soil pollution risk (Yao et al. 2010, Zhang et al. 2010, Zhao et al. 2020, He 2019, Jiang et al. 2020, Zhang et al. 2019, Li et al. 2008, Xie et al. 2020).

Dissolved organic carbon (DOC) refers to organic carbohydrates with relatively low molecular weights and simple structures. DOC can be dissolved in water and pass through a 0.45-μm microporous membrane. DOC is the most active component of soil organic carbon and the main carrier of many trace organic and inorganic pollutants (Kalbitz et al.

2000, Yang et al. 2020, Li et al. 2003, Ye 2020, Wang et al. 2020, Liu 2019a, Huang et al. 2015). DOC reacts directly with metal ions in water, soil, and sediment through a series of reactions such as ion exchange, adsorption, complexation, chelation, flocculation, and precipitation, thereby affecting the adsorption and desorption behaviors of heavy metals in soil (Yang & Zeng 2007, Wang 2016, Li 2017). Therefore, changes in the content of DOC inevitably lead to changes in the contents of heavy metals in soil. Studies have shown that DOC in soil affects the adsorption and improves the mobility of heavy metals in the soil (Jagadamma et al. 2014). Mahara et al (2007) reported that low-molecular-weight DOC in soil strongly affects the migration and transformation of Cd in the environment. Zhang (2020) found a strong correlation between dissolved heavy metals in the natural environment and the concentration of DOC. Liu (2019b) reported that DOC easily adsorbs and binds Cd in the soil. Adding DOC to the soil can increase the content of soil-exchanged Cd, and correlation analysis revealed a significant positive correlation between soil-exchanged Cd and soil DOC.

Previous research on soil DOC in China and elsewhere mainly focuses on the distribution and composition of DOC in soil, vegetation coverage and soil fertilization, and the adsorption, desorption, complexation, dissociation, precipitation, and

dissolution of soil heavy metal ions in the presence of DOC. However, the basic chemical equilibria along with the corresponding mechanisms, laws, and kinetic processes associated with the interactions between soil DOC and heavy metals have received less research attention. In this study, reclaimed soil in the subsidence area of Panyi Mine was chosen as the research object. Humic acid (HA) with the largest content of soluble humus was selected as a representative DOC molecule. The citric acid (CA), a small-molecule organic acid, served as a control. These two organic acids were used to prepare DOC solutions with different concentrations, which were used to determine the effects of DOC on the characteristics and kinetics of Cd adsorption and desorption in reclaimed soil. The results provide fundamental data for evaluating the Cd pollution status of reclaimed soil along with a scientific method for risk assessment and prediction. The findings also provide guidance for the rational application of organic materials.

MATERIALS AND METHODS

Study Area

The Panyi Mine Reclamation Area is located approximately 1.0 km east of the Panyi Mine, Panji District, Huainan City. A coal gangue hill is located on the southern side of the reclamation area. The reclamation area is formed by filling and rolling coal gangue in the coal mining subsidence area. The coal gangue is covered by clay with a design thickness of 100 cm. Tree species including poplar, privet, metasequoia, cedar, and Sichuan juniper were planted in the subsidence area after treatment. This area lies in a warm temperate semi-humid continental monsoon climate zone. The average annual temperature is 15.3°C, the highest temperature occurs in July, and the lowest occurs in January. The average annual precipitation is 926 mm.

Sample Collection and Pretreatment

Three sampling lines were established in the Panyi Mine Reclamation Area. A sampling point was located every 5 m on each sampling line, resulting in a total of 60 sampling points. At each sampling point, a soil heavy metal sampler was used to collect 0–20 cm of reclaimed soil. Each sample had a mass of at least 1 kg. After removing impurities, the soil sample was crushed and spread flat on an air-dried soil dish to dry. The sample was sieved using the quarter method, ground with an agate mortar, passed through a 100-mesh nylon screen, packed in a Ziploc bag, and labeled for use. Its physic-chemical properties were shown in Table 1.

Experimental Methods

As mentioned above, HA was selected as a representative DOC molecule, and CA served as a control. These two organic acids were used to prepare DOC solutions with different concentrations.

Adsorption of Cd in reclaimed soil under the influence of DOC: A reclaimed soil sample (1.000 g, passed through an 18-mesh screen) was weighed and placed in a 50-mL centrifuge tube. Twenty milliliters of $Cd(NO_3)_2$ solution with a Cd concentration of 0, 0.2, 0.4, 0.6, 0.8, 1, 1.5, 2, 2.5, 3, 4, 5, 8, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, or 80 mg.L^{-1} was added to the centrifuge tube followed by the addition of 1 mL of HA or CA solution with a concentration of 10 mg.L^{-1}. The centrifuge tube was then capped, shaken at 200 r.min^{-1} at a constant temperature of 25°C for 24 h, and then centrifuged at 4000 r.min^{-1} for 20 min. Finally, the supernatant was filtered through a 0.45-μm filter membrane, and one drop of 1% nitric acid was added to the filtrate followed by shaking for Cd determination. The concentration of Cd was analyzed by AAS (PerkinElmer AA800). The Cd adsorption capacity (Q_a, in mg.kg^{-1}) was determined based on the difference between the concentrations of Cd before and after adsorption:

$$Q_a = \frac{C_0 - C \times V}{m}, \qquad \ldots(1)$$

where C_0 is the concentration of Cd in the initial solution (mg.L^{-1}); C is the concentration of Cd in the equilibrium solution at adsorption equilibrium (mg.L^{-1}); V is the initial volume of liquid added (mL), and m is the mass of the sample (g). A control experiment was simultaneously carried out with 60 mg.L^{-1} HA and CA solution.

Desorption of Cd from reclaimed soil under the influence of DOC: The desorption experiment was carried out after the adsorption experiment was completed. The reclaimed soil after Cd adsorption was washed once with absolute ethanol to remove free Cd and placed at a constant temperature of 25°C for 1 h. Next, 20 mL of $NaNO_3$ solution (0.01 mol.L^{-1}) was added to the soil followed by covering and shaking at 200 r.min^{-1} and 25°C for 24 h. The solution was then centrifuged at 4000 r.min^{-1} for 20 min. Finally, the supernatant was filtered through a 0.45-μm filter membrane, and one drop of 1% nitric acid was added to the filtrate followed by shaking for Cd determination. The concentration of Cd was

Table 1: The physico-chemical properties of the soil.

Parameters	Values
PH	7.92±0.36
Organic matter (%)	1.06±1.08
Total N (%)	0.22±0.11
Available K (mg.kg^{-1})	127.28±16.26
Cd concentration (mg.kg^{-1})	0.80±0.22

analyzed by AAS (PerkinElmer AA800). The Cd desorption capacity (Q_d) was determined as

$$Q_d = \frac{C \times V}{m}. \qquad \ldots(2)$$

The Cd desorption rate (W) was calculated as

$$W\ (\%) = \frac{Q_d}{Q_a} \times 100\%. \qquad \ldots(3)$$

Data Analysis

The experimental data were statistically analyzed using SPSS 24.0 software. Plots were created using Origin8.0 software.

RESULTS AND DISCUSSION

Characteristics and Mechanism of Isothermal Cd Adsorption Under the Influence of HA and CA

As shown in Fig. 1, the two organic acids had different effects on Cd adsorption. As the concentration of Cd in the equilibrium solution increased, the adsorption capacity of reclaimed soil for Cd gradually increased. When the Cd concentration was low (0.2-10 mg.L^{-1}), the amount of adsorbed Cd was similar in the presence and absence of HA and CA. Thus, under low Cd concentrations, HA and CA had little effect on the ability of reclaimed soil to adsorb Cd. In contrast, when the Cd concentration was 15–80 mg.L^{-1}, the presence of HA and CA significantly reduced the adsorption of Cd by the reclaimed soil, and the amount of adsorbed Cd decreased as the concentration of added Cd increased. Thus, the addition of HA and CA significantly inhibited the adsorption of Cd by reclaimed soil. HA had a stronger inhibitory effect than CA, and the high organic acid concentration (60 mg.L^{-1}) produced a stronger effect than the low concentration (10 mg.L^{-1}). Different DOC components have been demonstrated to affect the migration of organic and inorganic contaminants in the soil in studies (Li et al. 2003, Zsolnay 1996, Gao et al. 2011, Lei et al. 2015, Bhattacharya & Osburn 2020). HA and CA contain a variety of active groups. In particular, HA contains abundant benzene rings, carboxyl groups, alcoholic hydroxyl groups, phenolic hydroxyl groups, and amino groups, which can undergo exchange, adsorption, and chelation reactions with heavy metals (Ma et al. 2020, Shan et al. 2015, Xu et al. 2008, Huang 2020). These sites compete with recovered soil for Cd adsorption, limiting the soil's ability to adsorb
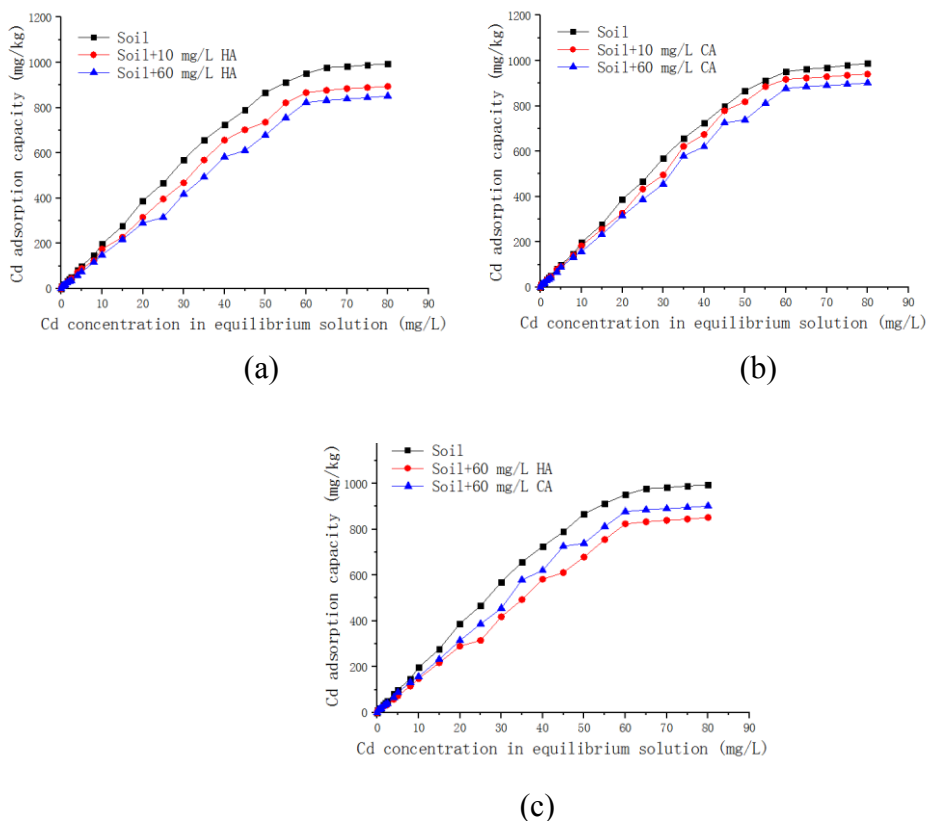

(a)

(b)

(c)

Fig. 1: Cd adsorption capacity vs. Cd concentration in solution in the presence and absence of HA and CA (60 mg.L^{-1}).
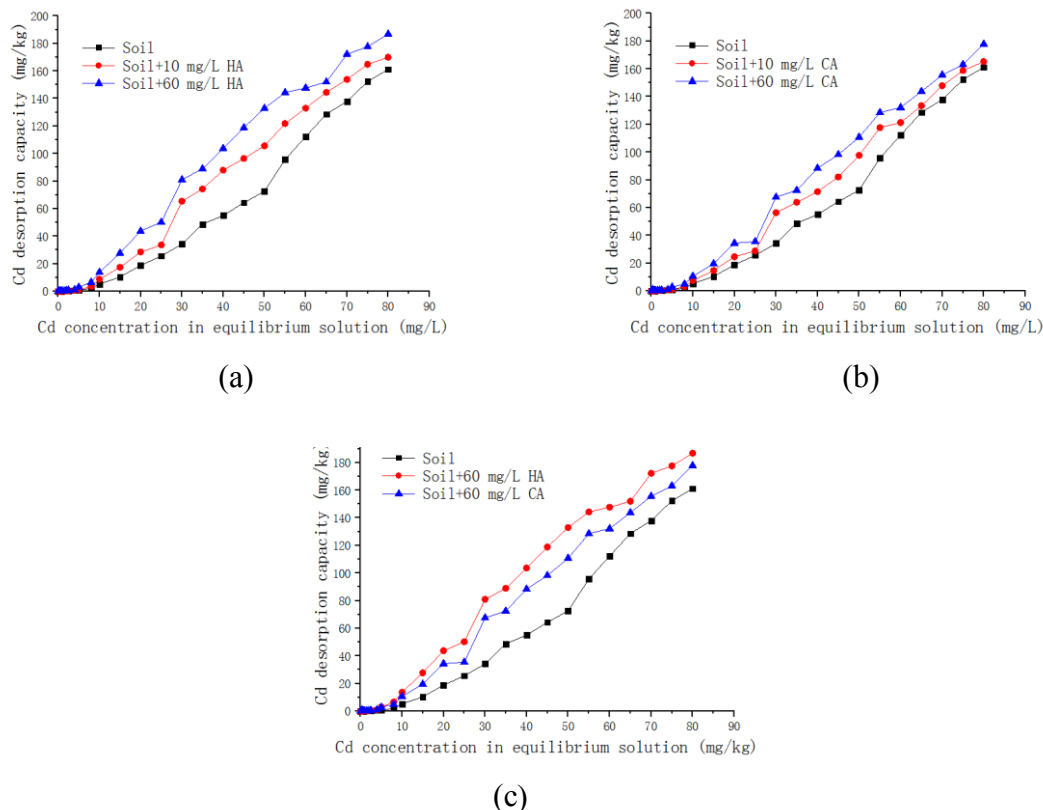
(a)

(b)

(c)

Fig. 2: Cd desorption capacity vs. Cd concentration in solution in the presence and absence of HA and CA (60 mg.L^{-1}).

Cd. The ionization of H+ in the oxygen-containing functional groups of HA and CA lowers the pH of the reclaimed soil solution, altering metal solubility and lowering the soil's Cd adsorption capacity (Li et al. 2018, Yang 2018, Yan 2019, Jagadamma et al. 2014). In addition, HA, which is rich in anionic groups such as phenolic, hydroxyl, and carboxyl groups, precedes Cd to occupy the soil seat sites through soil surface adsorption (He et al. 2020, Izosimova et al. 2020), thereby reducing the Cd adsorption capacity of reclaimed soil. This is consistent with previous results indicating that DOC has a significant inhibitory effect on Cd adsorption in soil (Chen & Chen 2002).

Characteristics and Mechanism of Isothermal Cd Desorption Under the Influence of HA and CA

As shown in Fig. 2, the ability of the two organic acids to desorb Cd was quite different. As the concentration of Cd in the equilibrium solution increased, the Cd desorption capacity of reclaimed soil gradually increased. When the Cd concentration was low (0.2–10 mg.L^{-1}), the amounts of desorbed Cd were similar in the presence and absence of HA and CA. Thus, under low Cd concentrations, HA and CA

had little effect on the ability of reclaimed soil to desorb Cd. When the Cd concentration was 15–80 mg.L^{-1}, the presence of HA or CA significantly increased the desorption of Cd in the reclaimed soil, and the amount of desorbed Cd increased as the concentration of added Cd increased. Due to the different physical and chemical properties of reclaimed soils, HA and CA had different effects on desorption capacity. HA had a stronger enhancement effect on Cd desorption, and high organic acid concentration (60 mg.L^{-1}) produced a larger effect than the low concentration (10 mg.L^{-1}).

As shown in Fig. 3, the desorption rate of Cd from reclaimed soil increased upon the addition of HA or CA. The Cd desorption rate of reclaimed soil increased on average by 5.71% and 3.21% upon the addition of high (60 mg.L^{-1}) and low (10 mg.L^{-1}) concentrations of HA, respectively. The Cd desorption rate increased by 3.48% and 1.94% in the presence of high and low concentrations of CA, respectively. The adsorption process of Cd is dominated by chemical bonding and electrostatic attraction. The activation energy required for adsorption is low, and the adsorption affinity is high. The desorption process of Cd is an ion exchange process. Organic ligands contained in organic acids can form soluble

organometallic complexes with Cd, thereby reducing the adsorption of Cd in reclaimed soil. In addition, the protons in HA and CA can activate Cd in reclaimed soil and promote its desorption (Xu et al. 2015).

Kinetics and Mechanism of Cd Adsorption and Desorption Under the Influence of HA and CA

As shown in Fig. 4, compared to the normal reclaimed soil, Cd adsorption was significantly reduced in the soil containing HA or CA. Under the influence of HA, Cd adsorption was reduced by 17.06%–18.80% (average = 18.18%). In the presence of CA, Cd adsorption in the reclaimed soil decreased by 12.58%–15.34% (average = 13.66%). Thus, HA reduced Cd adsorption in the reclaimed soil more obviously than CA. These results show that HA and CA changed the migration behavior of Cd in the reclaimed soil by affecting its adsorption.

The adsorption of Cd in reclaimed soil containing HA or CA followed the same pattern as it did in the absence of organic acids. Adsorption took place in two stages: a quick adsorption stage lasting 0–0.5 hours and a slower adsorption stage lasting 1–12 hours. The adsorption capacity of Cd in reclaimed soil increased substantially during the

fast absorption stage. Under the influence of HA, the Cd adsorption rate was 71.36%, corresponding to reductions of 15.04% compared to the normal reclaimed soil, respectively. In the presence of CA, the Cd adsorption rate was 72.15%, representing reductions of 11.80% compared to the normal reclaimed soil, respectively. In the slow reaction stage from 1–12 h, the amount of adsorbed Cd continued to increase, but the rate of increase was slower compared to in the fast reaction stage. Compared to in the normal reclaimed soil, the Cd adsorption capacity increased more slowly in the presence of HA or CA, and adsorption equilibrium was reached after 12 h. These results further demonstrate that HA and CA can inhibit the adsorption of Cd in reclaimed soil.

As shown in Fig. 5, under the influence of HA or CA, the Cd desorption capacity in reclaimed soil first decreased and then increased over time, revealing two desorption stages: a fast reaction stage from 0–2 h from the beginning of the experiment and a slow reaction stage from 2.5–12 h. In the first 2 h, desorption capacity decreased rapidly, reaching the lowest value at 2 h. In the subsequent slow reaction stage, the desorption capacity increased slowly, reaching equilibrium after approximately 12 h. Both HA and CA significantly increased the desorption capacity of Cd in reclaimed soil; HA increased the Cd desorption capacity by 28.51%–72.98% (average = 50.29%), while the effect of CA was weaker (increase in desorption capacity of 9.56%–42.67% with an average increase of 26.34%). These results demonstrate that HA and CA can alter the migration activity of Cd in reclaimed soil by affecting the desorption of Cd.

Adsorption and Desorption Characteristics and Mechanism of Cd in the Presence of Different Concentrations of HA and CA

As shown in Fig. 6, the two organic acids had different effects on the adsorption of Cd in the reclaimed soil, with HA having a stronger effect than CA. Within the range of organic acid concentrations examined in this study (0–80 mg.L^{-1}), the
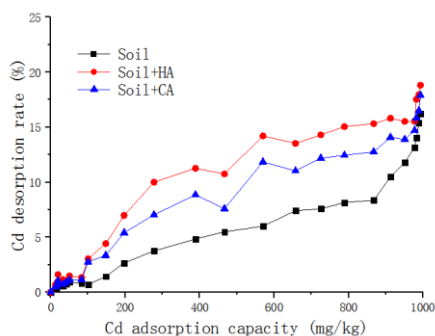

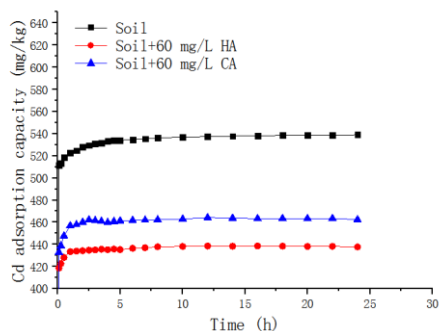Fig. 3: Cd desorption rate vs. Cd adsorption capacity for reclaimed soil in the presence and absence of HA and CA.

Fig. 4: Kinetic curves of Cd adsorption in reclaimed soil in the absence and presence of CA and HA.
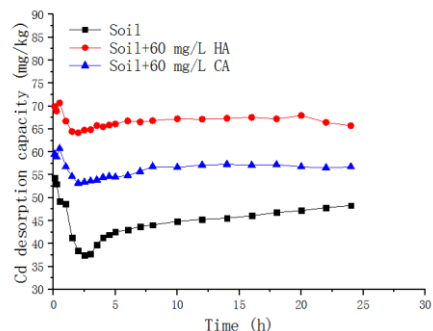

Fig. 5: Kinetic curves of Cd desorption from reclaimed soil in the presence and absence of CA and HA.
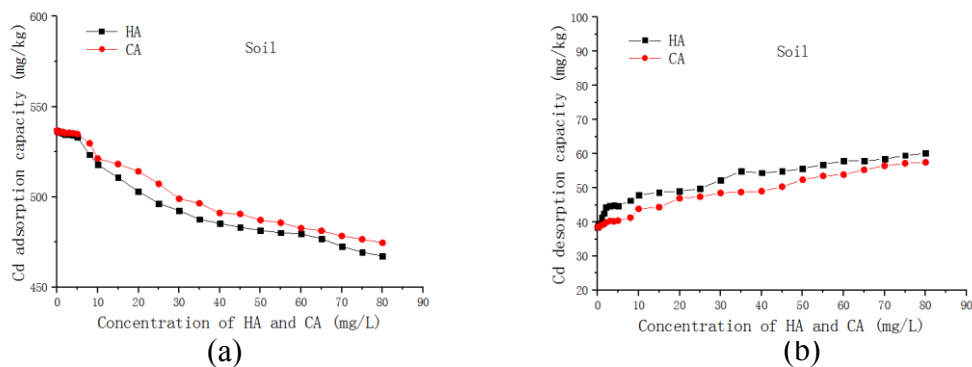
Fig. 6: Plots of Cd adsorption capacity (left) and desorption capacity (right) vs. HA and CA concentration.

adsorption capacity of Cd in the reclaimed soil gradually decreased with increasing HA or CA concentration. In the HA system, the adsorption rate of Cd in the reclaimed soil was reduced from the initial value of 95.02% to 80.60%; in the CA system, the adsorption rate of Cd decreased to 82.39%. The reclaimed soil was weakly alkaline (pH = 7.91). Since alkaline soil contains more clay minerals than acidic soil, the adsorption capacity for Cd in alkaline soil is relatively high. The addition of organic acids results in the complexation between organic ligands and Cd. The negative charge in the soil solution repels the negative charge on the soil surface (Wang 2012). In this case, the addition of organic acid reduced the adsorption capacity of Cd, and as the concentration of added organic acid increased, the adsorption capacity gradually decreased.

The two organic acids had different effects on the desorption of Cd in the reclaimed soil, with HA having a stronger effect than CA. As the concentration of organic acid increased within the tested range (0–80 mg.L^{-1}), the amount of desorbed Cd in the reclaimed soil gradually increased; that is, the organic acids promoted Cd desorption in the reclaimed soil. In the HA system, the desorption rate of Cd from the reclaimed soil increased from the initial value of 7.18% to 12.87%; in the CA system, the desorption rate increased to 12.12%.

Regression analysis revealed that the relationship between the concentration of organic acid (Y) and the Cd adsorption or desorption capacity (X) in reclaimed soil was best described by the logarithmic function $Y = a\ln X + b$, where a and b reflect the differences in the behaviors of HA and CA. The specific functions for Cd adsorption and desorption are shown in Table 2 and Table 3, respectively.

The established logarithmic function $Y = a\ln X + b$ is suitable for describing Cd adsorption and desorption in reclaimed soil under the influence of organic acids in the study area. The adsorption and desorption of Cd in reclaimed soil in the presence of varied organic acid concentrations can be calculated by varying the values of a and b.

CONCLUSION

The effects of organic acids on Cd adsorption and desorption in reclaimed soil are complicated. In this study, the addition of two representative organic acids, HA and CA, inhibited the adsorption of Cd in reclaimed soil, with HA having a stronger inhibitory effect than CA. Furthermore, both HA and CA increased Cd desorption in reclaimed soil, with HA being more effective than CA.

Table 2: Logarithmic regression equations for the relationship between HA and CA concentration (Y) and adsorption capacity (X).

Organic acid	Sample	Equation	R^2	n
HA	Soil	$Y = -28.3\ln X + 610.26$	0.9943	27
CA	Soil	$Y = -25.141\ln X + 607.59$	0.9937	27

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Table 3: Logarithmic regression equations for the relationship between HA and CA concentration (Y) and desorption capacity (X).

Organic acid	Sample	Equation	R^2	n
HA	Soil	$Y = 5.5037\ln X + 34.855$	0.9532	27
CA	Soil	$Y = 5.6876\ln X + 30.544$	0.9536	27

The adsorption and desorption kinetics of Cd in reclaimed soil could be divided into two stages: an initial fast reaction stage followed by a slow reaction stage. For Cd adsorption, the adsorption capacity of reclaimed soil increased rapidly during the fast reaction stage from 0–0.5 h. During the slow reaction stage from 1–12 h, the Cd adsorption capacity continued to increase but at a slower rate. The adsorption equilibrium was reached after 12 h. The adsorption rates of Cd in reclaimed soil were 15.04% and 11.80% lower in the presence of HA (60 mg.L^{-1}) and CA (60 mg.L^{-1}) than in the absence of organic acids, respectively.

For Cd desorption, the desorption capacity decreased rapidly in the fast reaction stage from 0–2 h. In the slow reaction stage from 2.5–12 h, the desorption capacity increased slowly with time and reached equilibrium after 12 h. In the presence of HA (60 mg.L^{-1}) and CA (60 mg.L^{-1}), the Cd desorption rate in reclaimed soil was 50.29% and 26.34% higher than in the absence of organic acids, respectively.

The relationship between the adsorption or desorption capacity of Cd and the concentration of organic acid was best described by the logarithmic function $Y = a\ln X + b$.

ACKNOWLEDGMENTS

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