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Evaluation of palygorskite for remediation of Cd-polluted soil with different water conditions

Jianrui Li^{1,2} \cdot Yingming Xu¹

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Abstract

Purpose The study aimed at comparing the effects of different water managements on soil Cd immobilization using palygorskite, which was significant for the selection of reasonable water condition.

Materials and methods Field experiment was taken to discuss the in situ remediation effects of palygorskite on Cd-polluted paddy soils, under different water managements, using a series of variables, including pH and extractable Cd in soils, plant Cd, enzyme activity, and microorganism number in soils.

Results and discussion In control group, the pH in continuous flooding was the highest under three water conditions, and compared to conventional irrigation, continuous flooding reduced brown rice Cd by 37.9%, and brown rice Cd in wetting irrigation increased by 31.0%. In palygorskite treated soils, at concentrations of 5, 10, and 15 $g kg^{-1}$, brown rice Cd reduced by 16.7, 44.4, and 55.6%; 13.8, 34.5, and 44.8%; and 13.1, 36.8, and 47.3% under continuous flooding, conventional irrigation, and wetting irrigation ($p < 0.05$), respectively. The enzyme activity and microbial number increased after applying palygorskite to paddy soils.

Conclusions Continuous flooding was a good candidate as water management for soil Cd stabilization using palygorskite. Rise in soil enzyme activity and microbial number proved that ecological function regained after palygorskite application.

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 \boxtimes Jianrui Li [jianrui-419@163.com](mailto:jianrui-163.com)

² Taiyuan Institute of Technology, Taiyuan 030008, China

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1 Introduction

As the industrial development and population growth, cadmium contamination of soils has turned into a trouble issue in China. The survey from 11 provinces done by Ministry of Agriculture of China found that 1.3×10^4 ha of farmlands has been polluted by cadmium (Zhu et al. [2004\)](#page-7-0). Cadmium is one of the most toxic pollutants in soils because of its persistence, toxicity, and potential for bioaccumulation. Cadmium harms the kidney and contributes to violence and some disorders of behavior and mental attitude (Zhou and Song [2004\)](#page-7-0). So cadmium has been prohibited by European Union's Restriction of Hazardous Substances Directive (European Union [2002](#page-6-0)). Rice plant, one of the main food crops in China, was capable of accumulating relatively high levels of cadmium from contaminated soils. As a consequence, high accumulation of Cd in rice grain would pose a potential hazard to human health via food chains (Méndez-Armenta and Ríos [2007](#page-7-0)).

A two-aspect tactics had been adopted to remediate metalcontaminated soils (Zhou et al. [2006;](#page-7-0) Cao et al. [2008\)](#page-6-0). The first aim is the in situ enhancement of heavy metal stabilization in soils, whereas the other is the ex situ extraction of metals from polluted soils. Immobilization remediation of metalcontaminated soils is a technology that can stabilize metals by applying amendments to soils, decreasing their bioavailability (Kumpiene et al. [2008;](#page-7-0) Raicevic et al. [2009](#page-7-0)). Among these amendments, natural clay minerals such as zeolite (Chen et al. [2000;](#page-6-0) Mahabadi et al. [2007](#page-7-0); Li et al. [2009](#page-7-0)), apatite (Raicevic et al. [2006](#page-7-0)), and sepiolite (Xu et al. [2009](#page-7-0); Xu et al. [2010;](#page-7-0) Sun et al. [2012](#page-7-0)) have been widely utilized for removal of trace elements from contaminated soils for their availability and low cost.

¹ Agro-Environmental Protection Institute, Ministry of Agriculture, Tianjin 300191, China

The investigations have shown that palygorskite was an effective amendment to reduce soil Cd uptake by plant (Liang et al. [2011](#page-7-0); Wang et al. [2011\)](#page-7-0). Furthermore, phytoavailability of Cd in acid soils has been proved to have marked distinctions among different water managements (Hu et al. [2010](#page-6-0)). However, technologies of palygorskite application combined with moisture management, used for metalcontaminated soils remediation, have rarely been reported so far. In addition, to evaluate the effects of management practices on soil quality and thus to prognosticate their consequences on environment, numerous researches have attempted to determine potential microbial parameter and soil enzyme activity as indicators (Garau et al. [2007](#page-6-0); Stępniewska et al. [2009](#page-7-0); Gao et al. [2010](#page-6-0)).

Guiyang from Hunan province is situated on upstream of Chong-ling Jiang, one of winding channels of Xiang Jiang. The local farmlands were contaminated by cadmium because of Pb and Zn smelting and mining. An in situ field-scale remediation was conducted in Guiyang. However, Pb, as one of two mostly concerned heavy metals in brown rice, was 50.8 mg kg⁻¹ in tested soils, which was lower than 80 mg/ kg (maximum permissible Cd concentration in soils by Chinese Soil Environmental Quality Standard-GB15618- 2008). Zn was not divided among the toxic elements. So cadmium was the only target pollutant in tested soils.

In the research, under different water conditions, the following experimental indexes were assessed: firstly, the effects of palygorskite on rice plant growth and Cd uptake, and secondly, the influences of palygorskite on pH, extractable Cd, enzymes activity, and microbial community in soils.

2 Materials and methods

2.1 Amendment property

The palygorskite material, a naturally occurring clay mineral with 64.4% SiO_2 , 20.5% MgO, 10.4% Al₂O₃, 1.5% Na₂O, and 1.2% CaO, was supplied by a material manufactory. It had a high cation exchange capacity (CEC = 0.45 meq g^{-1}) and pH at a point of zero charge ($pH = 9.53$).

2.2 Soil characterization and plant culture

Soil materials having been passed through a 20-mesh sieve were prepared for physicochemical property analysis. Soils examined in the study were classified as Red Earth by Chinese Soil Taxonomy (Gong [1999\)](#page-6-0). The selected properties of soil samples are listed in Table [1](#page-2-0). We made application of palygorskite to paddy soils at 0.50 kg m^{-2} (0.5%), 1.00 kg m⁻² (1.0%), 1.50 kg m⁻² (1.5%), and a no-amendment treatment (0%). Palygorskite was mixed into topsoil (0–20 cm). Soil water management was carried out using following methods, involving continuous flooding (5–7 cm surface water during the whole growth period of rice plant), conventional irrigation (moist soil surface during the late tillering state and grain filling stage, and 5–7 cm surface water during the other growth stages of plant), and wetting irrigation (moist soil surface during the whole growth period of plant, about 70% of field water-holding capacity). There were totally 12 experimental treatments (4×3) and 36 plots (each measuring $5 \text{ m} \times 6 \text{ m}$).

The rice plant $(TY-272, \text{ ordinary rice cultivar})$ transplanting was completed 30 days after palygorskite application, which was consistently with local conventional practice. A 250-g composite topsoil sample was collected in each plot using five-point sampling and then air-dried for chemical analysis. After 120 days of growth, rice plants were harvested and flushed with tap water, then rinsed three times with deionized water. The plant was divided into rice straw and brown rice, then dried to a constant weight at 65 °C in oven; plant sample was smashed with a stainless mill and passed through a 0.25-mm sieve before physicochemical analysis.

2.3 Analytic methods

2.3.1 pH and extractable Cd

The determinations of pH for top-soil of plots were done using automatic multi-function pH analyzer (FJA-6) after rice transplant and before plant harvest. The phyto-available Cd in soil was estimated using three different extraction solutions. Five grams of soil samples was dispersed into 25 ml of 0.025 M and 0.1 M HCl solutions and shaken for 60 min (Kikuchi et al. [2008\)](#page-6-0). Additionally, 10.0 g soil samples was dispersed into 50 ml of 0.01 M CaCl₂ solution and shaken for 120 min. All the supernatants of the extractions were collected by centrifugation at 4300 rpm.

2.3.2 Total Cd

The plant and soil samples were treated with mixtures of $HNO₃-HClO₄$ and $HNO₃-HF-HClO₄$, respectively. The resulting solutions were filtered with paper filter and diluted to 100 ml with high purity water. The Cd concentrations in solutions were detected using ICP-AES (ZEEnit 700P, Analytikjena, Germany). Bush leaf (GBW07603, China) materials, as certified reference materials, were used to evaluate the accuracy of digestion procedure and subsequent analysis, and the accuracy obtained using reference material was 3.1% (RSD).

2.3.3 Soil enzyme activity

Catalase activity was analyzed using H_2O_2 as substrate, shaking for 20 min, then the solution was titrated by $KMnO₄$

Table 1 The basic physical and chemical properties of tested soil

(Stępniewska et al. [2009\)](#page-7-0). Phosphatase activity was measured by sodium bis(p-nitrophenyl) phosphate as substrate, incubating at pH 14.0 and 30 °C for 10 min, and residual substrate was determined by colorimetric method. Protease activity was determined using gelatin solution as substrate, incubating at 30 °C for 24 h, and formed amino acid was measured with colorimetric method (Bhattacharyya et al. [2008](#page-6-0)).

2.3.4 Soil microorganism population

Microorganism quantity was evaluated by dilution plate technology (Gao et al. [2010](#page-6-0)). Bacteria: beef extract 5 g, peptone 10 g, NaCl 5 g, distilled water 1000 ml, agar 17 g, pH 7.2–7.4. The bacterium-mixing plate method was used and 10^{-5} – 10^{-7} dilutions of soil samples were used as inoculums, and the colonies were counted after incubation at 30 °C for 3 days. Fungi: glucose 10 g, K_2HPO_4 1.0 g, $MgSO_4$ ·7H₂O 0.5 g, distilled water 1000 ml, agar 17 g, pH 4.0–5.0. The fungi-mixing plate method was used and 10^{-1} – 10^{-3} dilutions of soil samples were used as inoculums, and the colonies were reckoned after incubation at 30 °C for 3 days. Actinomycetes: soluble starch 20 g, KNO3 1.0 g, K2HPO4 0.5 g, MgSO4·7H2O 0.5 g, NaCl 0.5 g, FeSO4·7H2O 0.01 g, distilled water 1000 ml, pH 7.2–7.4. The actinomycete-mixing plate method was used and 10^{-3} – 10^{-5} dilutions of soil samples were used as inoculums, and the colonies were counted after incubation at 30 °C for 5 days.

2.4 Statistical analysis

All treatments were replicated three times in the research. All data were represented as means \pm standard deviations. Analysis of variance was performed with SAS 8.5. When a significant ($p < 0.05$) difference was observed between treatments, multiple comparison was made by LSD test. All graphs were created using Origin 8.0 software.

3 Results

3.1 Plant growth and Cd uptake

The inhibitory effects on crop growth by heavy metals could act as an index of its toxicity to plants, and increase in plant biomasses indicated a decline in biological toxicity. As revealed in Fig. 1, in control soils, in comparison to conventional irrigation, brown rice production reduced by 11.5 and 23.7% in continuous flooding and wetting irrigation

 $(p < 0.05)$. The largest percentage increase of brown rice productions reached 4.9, 7.3, and 7.6%, at palygorskite concentration of 15 g kg^{-1} , in continuous flooding, conventional irrigation, and wetting irrigation, which was probably due to reducing Cd inhibition of plant growth $(p < 0.05)$.

The contents of Cd in brown rice and rice straw are shown in Fig. [2.](#page-3-0) Generally, Cd concentrations of aboveground parts of plant declined in order rice straw > brown rice. In contrast, compared with conventional irrigation, continuous flooding reduced brown rice Cd by 37.9%, and brown rice Cd in wetting irrigation increased by 31.0% ($p < 0.05$), brown rice Cd in long-term flooding was below 0.2 mg kg^{-1} (fresh weight, national allowable limit for Cd). The rice straw Cd showed the same changing trend with brown rice Cd, suggesting that long-term flooding reduced soil Cd transfer to rice plant. In palygorskite treated soils, at concentrations of 5, 10, and 15 g kg−¹ , brown rice Cd reduced by 16.7, 44.4 and 55.6%; 13.8, 34.5, and 44.8%; and 13.1, 36.8, and 47.3% under continuous flooding, conventional irrigation, and wetting irrigation $(p < 0.05)$, respectively, which revealed that the lower palygorskite addition could obtain the same brown rice Cd from wetting irrigation to continuous flooding. Obviously, as the increasing palygorskite applied concentration, the increment in decreasing amplitude of brown rice Cd declined gradually. Furthermore, compared to corresponding controls, at the same palygorskite content, decreasing amplitude of brown rice Cd was higher in continuous flooding than in conventional irrigation and wetting irrigation. It was worthwhile to note that the same variety laws were found for rice straw Cd.

Fig. 1 The biomasses of rice plant under different treatments. Vertical *bars* represent means \pm standard deviations. Same letters above bars are not significantly different using LSD test ($p < 0.05$; $n = 3$)

Fig. 2 The Cd concentrations of rice plant under different treatments. Vertical bars represent means \pm standard deviations. Same letters above *bars* are not significantly different using LSD test ($p < 0.05$; $n = 3$)

Table 2 The soil pH and extractable Cd under different treatments

Hence, continuous flooding management helped to implement soil Cd immobilization using palygorskite.

3.2 pH and extractable Cd

The pH was one of the chief factors governing heavy metal bioavailability in acid soils. As reflected in Table 2, for the same soil treatment, the pH after rice transplant was much lower than that before plant harvest. Before plant harvest, in contrast, because of gradual consumption of H^+ (chief reducing force in acid soils) during the long-term process, pH in continuous flooding was the highest among three water managements. Soils used in this study were acid, and pH increased with a rise in levels of palygorskite addition. Before harvest, increase in pH at a serial concentration of palygorskite ranged from 0.49 to 1.05, from 0.40 to 0.98, and from 0.50 to 1.00 units under continuous flooding, conventional irrigation, and wetting irrigation ($p < 0.05$), respectively. This is due to palygorskite alkaline properties, depicted in Sect. [2.1](#page-1-0).

As shown in Table 2, in control soils extracted by 0.025 M HCl–0.1 M HCl–0.01 M CaCl₂, compared to conventional irrigation, continuous flooding reduced extractable Cd by 17.4 to 18.8%, and extractable Cd in wetting irrigation increased by 12.5 to 13.0% ($p < 0.05$). The higher pH in continuous flooding caused a rise in variable negative charges of soils and an increase of Cd adsorption onto colloids, ultimately resulting in lower extractable Cd in soils. At palygorskite contents of 5, 10, and 15 $g kg^{-1}$, 0.025 M HCl extractable Cd was reduced by 13.2 to 44.7%, 10.9gorskite alkaline properties, depicted i to 36.9%, and 11.5 to 36.5% in continuous flooding, conventional irrigation, and wetting irrigation; 0.1 M HCl extractable Cd reduced by 10.8 to 43.5%, 8.9 to 32.1%, and 11.1 to 31.7%, and 0.01 M CaCl₂ extractable Cd

The same letters after means in the column are not significantly different using LSD test ($p < 0.05$; $n = 3$). The same below

reduced by 15.4 to 53.8%, 12.5 to 43.7%, and 16.6 to 44.4% $(p < 0.05)$. Plainly, with the rising palygorskite addition in soils, the increment in decreasing amplitude of soil extractable Cd diminished progressively. Then again, compared to corresponding controls, at the identical palygorskite concentration, the decreasing amplitude of extractable Cd was higher in continuous flooding than in conventional irrigation and wetting irrigation, indicating that the remediation effects for polluted acid soils would become better when the extractable Cd were at lower level. The lower H^+ content in long-term flooding soil relieved the rivalry for absorption sites of palygorskite between H^+ and Cd^{2+} . Furthermore, continuous flooding propelled hydrolysis and hydroxylation of Cd^{2+} because of lower $H⁺$ concentration, which conduced to Cd adsorption onto palygorskite. The brown rice Cd was positively related with 0.025 M HCl extractable Cd ($R^2 = 0.68$, $p < 0.05$), 0.1 M HCl extractable Cd ($R^2 = 0.43$, $p < 0.05$), and 0.01 M CaCl₂ extractable Cd ($R^2 = 0.55$, $p < 0.05$). Obviously, 0.025 M HCl extractable Cd was a reasonable scheme for the evaluation of Cd phytoavailability in soils. Negative correlation was observed between pH and rice straw Cd ($R^2 = 0.51$, $p < 0.05$) and brown rice Cd ($R^2 = 0.47$, $p < 0.05$).

3.3 Enzyme activity

The enzyme activity, an important member of bioactivity in soils, plays a vital role in mineralization of organic matters and decomposition and formation of humus in soils (Zhang and Wang [2006](#page-7-0)). Catalase could decompose H_2O_2 into O_2 and H_2O , having links with metabolic activity of aerobic organisms (Stępniewska et al. [2009\)](#page-7-0). In control soils, as depicted in Fig. 3a, catalase activity was lower in continuous flooding than in conventional irrigation ($p < 0.05$), and there was no significant difference between conventional irrigation and wetting irrigation. Catalase activities in amended soils, with the increasing percentage of palygorskite, showed no remarkable variation under different water managements. Phosphatase is an important impetus of organophosphate hydrolysis in acid soils (Bhattacharyya et al. [2008](#page-6-0)). In control group, as shown in Fig. 3b, compared to conventional irrigation, phosphatase activity in continuous flooding reduced by 8.9% and that in wetting irrigation increased by 13.1%. Phosphatase activities in soils amended with palygorskite, at concentrations of 5, 10, and 15 g kg^{-1} , increased by 5.7 to 13.3%, 5.2 to 12.9%, and 6.9 to 14.5% under continuous flooding, conventional irrigation, and wetting irrigation. Protease activity is closely connected with organic matter and nitrogen in soils (Bandick and Dick [1999](#page-6-0)). As revealed in Fig. 3c, in untreated soils, protease activity declined in order of wetting irrigation > conventional irrigation > continuous flooding, and palygorskite addition enhanced its activity in soils ($p < 0.05$). The statistical analysis shown that pH was positively correlated with enzyme activity ($R^2 = 0.47$, $p < 0.05$).

Fig. 3 The activities of soil enzymes under different treatments. Vertical bars represent means \pm standard deviations. Same letters above bars are not significantly different using LSD test ($p < 0.05$; $n = 3$)

3.4 Microbial population

Microbial populations, as an impetus of nutrients transformation in soils, could keep community stability in a fluctuant surrounding (Aciego Pietri and Brookes [2008](#page-6-0)). As shown in Table [3](#page-5-0), in control group, amounts of microorganisms, including bacteria, fungi and actinomycete, increased in sequence of

Table 3 The soil microbial properties under different treatments

Water management	Palygorskite	Bacteria $(10^7 \text{ CFU soil g}^{-1})$	Fungi $(10^5$ CFU soil g^{-1})	Actinomycete $(10^6$ CFU soil g^{-1})
Continuous flooding	θ	$1.47 \pm 0.21e$	1.73 ± 0.38 bc	2.82 ± 0.52 f
	0.5%	1.61 ± 0.33 d	$1.38 \pm 0.33c$	3.66 ± 0.95 de
	1%	1.78 ± 0.51 cd	0.88 ± 0.13 de	$3.91 \pm 1.03d$
	1.5%	$1.91 \pm 0.22c$	$0.72 \pm 0.12e$	4.15 ± 1.13 cd
Conventional irrigation	θ	1.81 ± 0.11 cd	2.13 ± 0.38 ab	3.32 ± 0.88 e
	0.5%	2.02 ± 0.13 bc	1.78 ± 0.18 bc	$4.41 \pm 0.75c$
	1%	2.23 ± 0.27	1.15 ± 0.23 cd	4.78 ± 1.02 bc
	1.5%	2.36 ± 0.21 ab	$1.01 \pm 0.15d$	5.01 ± 0.98 b
Wetting irrigation	θ	2.08 ± 0.47 bc	$2.21 \pm 0.77a$	$3.77 \pm 1.21d$
	0.5%	2.34 ± 0.67 ab	$1.94 \pm 0.93b$	5.16 ± 1.37 b
	1%	$2.62 \pm 0.57a$	$1.31 \pm 0.37c$	5.63 ± 1.58 ab
	1.5%	$2.73 \pm 0.88a$	1.12 ± 0.33 cd	$5.92 \pm 1.73a$

continuous flooding < conventional irrigation < wetting irrigation. In palygorskite treated soils, the quantities of bacteria and actinomycete were positively correlated with amendment addition under different water managements, but the fungi amount was inhibited after applying the palygorskite to soils. The pH was positively correlated with bacteria ($R^2 = 0.55$, $p < 0.05$) and actinomycete ($R^2 = 0.50$, $p < 0.05$). The negative relation was found between pH and fungi ($R^2 = 0.43$, $p < 0.05$).

4 Discussion

The clays decrease heavy metal bioavailability, which were caused by metal precipitation and complexation onto minerals, metal diffusion into lattice of minerals, and metal substrate retention, namely electrostatic interaction between metal and surface groups (Zhou et al. [2006](#page-7-0); Xu et al. [2009\)](#page-7-0). The positive influences of amendments on plant growth could be attributed to lower Cd bio-toxicity in soils. As shown in Sect.

Fig. 4 Plot of the combination of continuous flooding and in situ chemical immobilization by palygorskite

[3.1,](#page-2-0) increase in biomasses in polluted soils amended with palygorskite was observed, and plant Cd content declined after clay application. The similar result was obtained in other research that natural clay addition reduced the aboveground Cd concentration of rice plant under different treatments (Han et al. [2014](#page-6-0)). The exchangeable Cd declined and carbonatebound Cd rose significantly in sepiolite and bentonite treated paddy soils, which led to lower Cd bioavailability in amended soils (Sun et al. [2016\)](#page-7-0). In another investigation, Liang et al. [\(2014\)](#page-7-0) also found the application of sepiolite and palygorskite shifted soil Cd species from exchangeable Cd to residual Cd, and the TCLP and NH4OAc extractable Cd concentration decreased after applying above mentioned clays to rice fields. Similarly, in the present study, the significant decrease in the concentrations of HCl and CaCl₂ extractable Cd was observed by applying the palygorskite, indicating its effect on reducing soil Cd environmental risk.

The mobility of heavy metals increases with decreasing pH for lower chemical immobilization onto soil colloids

(Kumpiene et al. [2008](#page-7-0); Xu et al. [2010;](#page-7-0) Wang et al. [2011](#page-7-0)). Negative correlation was found between pH and soil Cd extractable concentration ($p < 0.05$), and significantly negative correlations were observed between pH and Cd concentrations in aboveground parts of rice plant ($p < 0.05$) (in Sect. [3.2\)](#page-3-0), consistent with the research (Wang et al. [2011](#page-7-0); Sun et al. [2012\)](#page-7-0).

The enzyme activity has been suggested as indicator for evaluating adverse effects of contaminants on soil ecosystem (Zhang and Wang [2006](#page-7-0); Cang et al. 2009). The lower phosphatase and protease activities in control soils indicated a poor ecological function, whereas the increased values observed in palygorskite treated soils proved that soil bioactivity rose (in Sect. [3.3](#page-4-0)). The higher enzyme activities in amended soils with respect to control showed that phosphatase and protease activities were influenced by extractable Cd and pH. However, other researchers found that enzyme activities in metalcontaminated soils were inhibited after amendment application (Garau et al. 2007), and the differences in soil types and heavy metal concentrations may be primary causes of abovementioned distinctions. The changes in microbial populations could precede fluxes in soil physicochemical characteristics, which can potentially serve as early signals of soil degradation or improvement (Aboim et al. 2008). Microbial populations were impressible to soil management mode. Microbial population changes, as described in Sect. [3.4,](#page-4-0) showed that soil bioactivity increased after palygorskite addition. Researchers also demonstrated that, compared to control group, basal soil respiration, dehydrogenase, acid and alkaline phosphatase, β-glucosidase, and arylsulfatase activities in sepiolite treated soils showed a remarkable increase because of lower soil Cd leachability and phytoavailability (Abad-Valle et al. 2016).

The significant correlation was observed between pH and biomass C (microorganism number) demonstrating pH role in stimulating microbial biomass rise in soils (de Mora et al. 2005). In the research, as shown in Sects. [3.3](#page-4-0) and [3.4](#page-4-0), the pH correlated positively with protease activity and bacteria community, but the amount of fungi has a negative correlation with phosphatase activity and actinomycete community, suggesting the importance of pH increase on microbial properties and soil enzymes activities. A similar research result was attained that bacterium number and urease activity correlated positively with pH (Sun et al. [2013](#page-7-0)).

5 Conclusions

Under different water conditions, palygorskite application decreased Cd uptake by rice plant and improved the bioactivity of Cd polluted paddy soils. Continuous flooding management promoted soil Cd immobilization using palygorskite. The enhancive values of enzyme activity and microbial number demonstrated that ecological function regained after applying

palygorskite to paddy soils. As shown in Fig. [4,](#page-5-0) continuous flooding practice and clay application should be integrated to develop safe agricultural production in Cd-contaminated rice fields.

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