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Enhanced flocculation and sedimentation of trace cadmium from irrigation water using phosphoric fertilizer



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HIGHLIGHTS

GRAPHICAL ABSTRACT

- The use of K₂HPO₄ as a flocculants for the removal from irrigation water of Cd combined with SS was investigated.
- The flocculation of Cd-SS binary system was attributed to the charge neutralization and electric double layer compression
- K₂HPO₄ enhanced the binding behaviors of cations towards to SS
- Oxidation of Fe²⁺ to Fe³⁺ by K₂HPO₄ was founded



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ABSTRACT

Suspended substrate (SS) in natural waters controls the interaction, transportation, and biological effects of heavy metals in water bodies. The large amount of cadmium (Cd) carried by SS is an important source of Cd pollution in irrigation water. In this study, a novel procedure to remove trace Cd coexisting with SS from irrigation water by fertilizer flocculation was investigated. Four common fertilizers (K2HPO4, (NH4)2HPO4, KH2PO4, and K2SO4) were used as flocculants. Batch experiments with various fertilizers revealed that the removal efficiency followed the order: $K_2HPO_4 > (NH_4)_2HPO_4 > K_2PO_4 > K_2SO_4$. When levels of K_2HPO_4 higher than 0.75 g/L were applied, the total Cd decreased from 20 to 3.8 µg/L after 12 h of flocculation, i.e., the removal efficiency reached 80%. The mechanism analysis(Zeta potential, multi-element analysis) results demonstrated that the aggregation of SS and its sedimentation by K_2 HPO₄ were due to the combined effect of the oxidation of Fe²⁺ to Fe³⁺ and cation binding behavior towards SS, as a result of charge neutralization and electric double layer compression. This method of Cd removal from irrigation water is simple and has the potential to be applied in agricultural production.

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1. Introduction

Public health safety events triggered by the effects of cadmium (Cd) pollution on rice production have aroused public concern about the

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current status of soil contamination in China. In recent decades, due to the anthropogenic pressure on mineral exploitation, ore extraction, and refining activities, harmful heavy metals have been discharged into farmlands via agricultural and aquaculture pond run-off, waste water, and industrial effluents (Lei et al., 2015; Wang et al., 2008; Zeng et al., 2015). A nationwide survey of soil pollution led by the Ministry of Environmental Protection (MEP) and the Ministry of Land and Resources (MLR) of the People's Republic of China in 2014 revealed

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that >16% of soils are contaminated by heavy metals, with the figure being 19% for farmland soils. It is estimated that approximately 26 million ha of farmland are polluted by Cd (Peng et al., 2016; Zhao et al., 2015). Contaminated arable soils have already seriously impeded agricultural development and threaten the safety of agricultural products (Du et al., 2013; Lei et al., 2015), especially rice production in Hunan province, southern China, where Cd pollution is a concern (Wang et al., 2008; Zeng et al., 2015). It is believed that irrigation waste water is the main source of Cd pollution in Hunan province (Zhao et al., 2015).

Irrigation water usually contains large amounts of suspended substrate (SS). This originates from continental weathering (Fitzsimons et al., 2011), coastal erosion, in-situ biogeochemical processes, atmospheric deposition, and industrial discharges, which are ubiquitous in natural water environments (Turner and Millward, 2002). The SS plays an important role in geochemical cycling in natural water, determines the fate of trace pollutants and provides a crucial link for chemical constituents between water bodies, bed sediments, and food chains (Gustafsson and Gschwend, 1997; Turner and Millward, 2002). The SS has a heterogeneous nature due to its complex composition (clay minerals, organic matter, and iron and manganese oxides), and thus flocculation and sedimentation could lead to the significant removal of trace metals due to the fact that colloidal matter has a large sorption capacity (per unit mass), resulting from its large specific surface area and abundant functional groups (Jain and Ali, 2000; Jain and Sharma, 2002). Trace heavy metals, such as Cd, once released into the aquatic environment, will transfer to sediments by adsorption onto SS and subsequent sedimentation (Zwolsman et al., 1993). Up to 82% of dissolved Cd becomes associated with sediments as a result of this process (Braungardt et al., 2009; Waeles et al., 2008). Cadmium combined with SS is resistant to gravitational settling, due to the fine particle size and negative charge. Therefore, without interference from external factors, Cd will remain suspended in the water column for a long period of time. In colloid chemistry, the classical Derjaguin, Landau, Vervey, and Overbeek (DLVO) theory was proposed to describe the stability of SS (Li et al., 2014; Liu et al., 2007). It is claimed that electrostatic repulsive forces and van der Waals forces are the two main factors leading to the flocculation and dispersion of the SS. The mutual contact between the particles depends mainly on the attractive energy of the Van der Waals forces. When the electrostatic repulsion between the SS particles is larger than the attractive Van der Waals forces, the SS cannot be cohesive. Hence, according to DLVO theory, if almost all the impurities are negatively charged, SS destabilization can occur (Duan and Gregory, 2003).

To meet the needs of agricultural production (rice production), irrigation water has the following characteristics. (1) Large volumes of water are needed during rice growth and the irrigation period is not continuous. (2) There are trace concentrations of Cd (below 20 μ g/L) in irrigation water. (3) During treatment processes, toxic and hazardous substances cannot be used and the pH cannot be changed significantly. The existing technologies are hard to meet the actual needs of irrigation water purification due to the complicated composition of irrigation water. Commonly used methods such as adsorption are susceptible to pH interference; the presence of coordination compounds could be interfering with chemical precipitation. The characteristics described above increase the difficulty of removing Cd from irrigation water. Moreover, because Cd is often present in complex forms, it is very difficult to achieve complete removal from irrigation water by means of a single technology. The amount of Cd interacting with SS is much higher than the amount of water-soluble Cd in irrigation water (Braungardt et al., 2009; Waeles et al., 2008); therefore, the removal of SS can effectively decrease the Cd concentration in irrigation water.

Many methods have been proposed for the removal of SS from waste water, including filtration (Yue et al., 2015), chemical precipitation (Rakesh et al., 2014), and bio-flocculation (Schlesinger et al., 2012). Considering the characteristics of Cd-polluted irrigation water when it interacts with SS, flocculation and precipitation may be excellent

methods to remove both the SS and Cd. It has been reported that SS and other impurities can be effectively removed from wastewater through charge neutralization and enmeshment by amorphous metal hydroxide precipitates (El Samrani et al., 2008). Furthermore, commercial coagulants, such as aluminum (Al) (Bo et al., 2011), ferrous sulfate, poly-titanium tetrachloride (Zhao et al., 2013), and polyacrylamide (Wei et al., 2014), are already widely used in conventional treatments (El Samrani et al., 2008). However, it is generally believed that traditional coagulants are not capable of dealing with Cd-polluted irrigation water, due to its complicated chemical consumption, non-biodegradable end-products, and sludge disposal problems.

In this study, the use of phosphoric fertilizer as a flocculants for the removal of Cd combined with SS from irrigation water was investigated. The chemical forms of Cd and the physical characteristics of irrigation water were thoroughly analyzed. The mechanism of SS flocculation under the influence of phosphoric fertilizer was discussed.

2. Material and methods

2.1. Preparation of irrigation water

In this study, to simulate irrigation water we used sediments and river water sampled from the Hunan Agricultural University (Changsha, China) stretch of the Liuyang River (N28°18'42.30", E113°10'32.81") in September 2015. The sediments were air-dried outside in the shade, and were then ground to pass through a 100-mesh 0.15-mm standard sieve in order to remove rock fragments and roots. The river water was filtered through medical gauze to remove hydrophytes (phytoplankton) and large-scale impurities, and then stored in a refrigerator at 5 °C. Irrigation stock solutions were prepared by dispersing 5.0 g of air-dried mud in river water, followed by 30 min of sonication in a water bath (KQ5200, KunShan Ultrasonic Instruments Co., Ltd. China). A stock solution of Cd (200 mg/L) was diluted ($0.55 \text{ g of Cd}(NO_3)_2 \cdot 4H_2O$ (GR pure) into 1000 mL of 1% HNO₃), and then kept in a refrigerator at 5 °C. Subsequently, the desired concentration of Cd (20 μ g/L) was obtained by serial dilution. Finally, the irrigation water (Cd 20 μ g/L) was stored in a polyethylene bottle for at least 72 h. These stock solutions were kept at 5 °C in the dark, with sonication again 30 min prior to the preparation of test solutions.

2.2. Batch experiments

Batch flocculation and sedimentation experiments were performed in 100-mL colorimetric tubes (Bomex, Beijing, Co., Ltd., China) containing increasing amounts of phosphoric fertilizer and 100 mL of irrigation water spiked with the Cd mixture to a final concentration of 20 µg/L. Irrigation solutions (100 mL with 20 µg/L Cd) were treated with di-potassium phosphate (K₂HPO₄), mono-potassium phosphate (KH₂PO₄), and di-ammonium hydrogen phosphate ((NH₄)₂HPO₄) with the concentrations of 0.75, 1.49, 2.25, and 3.00 g/L. The concentration of fertilizers used here was selected for better investigating the removal behaviors. These solutions were thoroughly sonicated for 30 min and then left to stand for 72 h. To determine the SS content and Cd removal at various time intervals, an individual tube was sampled successively over a series of time intervals (1, 2, 6, 12, 24, 48, and 72 h). All tests were run in triplicate. Five-milliliter samples were taken from the tops of the colorimetric tubes and the absorbance at 660 nm was measured by UV-VIS spectroscopy (UV-1780, Shimadzu, Japan) to determine the change in the SS content over time. The remaining solutions were then completely digested by adding 3 mL of concentrated HCl and 1 mL of concentrated HNO₃, and heating at 100 °C for 3-5 h in a water bath. The Cd concentration was subsequently determined by graphite furnace atomic absorption spectroscopy (GFAAS: GFA-6880, Shimadzu). At the each time intervals, the pH (PHS-3C, INESA Instrument Co., China) and electrical conductivity (EC: DDSJ-319L, INESA Instrument Co.) was also measured in situ.

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