Effect of divalent manganese ions on the roxarsone removal efficiency from aqueous solution with iron oxide

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ABSTRACT

In this study, the effect of Mn(II) on the roxarsone (ROX) removal efficiency by iron oxide was systematically investigated. ROX removed by iron oxide obeyed pseudo-second-order non-linear kinetic model with the addition of Mn(II), and the removal rate constant normalized by specific surface area was calculated to be $4.5 \times 10^{-3} \text{ g m}^{-2} \text{ h}^{-1}$, which was 4.8 times than that in the absence of Mn(II). Based on a series of experimental and characterization results, we found that Mn(II) could not be oxidized to Mn(III) in the presence of iron oxide, and the enhanced ROX removal process was attributed to the promoted ROX adsorption process. Further zeta potential results suggested that the addition of Mn(II) increased the isoelectric point of iron oxide from 5.3 to 7.5, which provided positive adsorption site favoring adsorption process of the negative charged arsenic functional group in ROX. This investigation provided fundamental understanding for the effect of Mn(II) on the removal efficiency of ROX by iron oxide.

Keywords: Divalent manganese ions; Roxarsone; Iron oxide; Removal mechanism

1. Introduction

Aromatic organoarsenicals, such as roxarsone (3-nitro-4-hydroxyphenylarsonic acid, ROX) and p-arsanilic acid (4-aminophenylarsenic, p-ASA), used as feed additive, has been widely applied for coccidiosis control and feed conversion promotion for decades [1]. However, most of these organoarsenic compounds little metabolized in the poultry or swine bodies, and almost excreted without transformation through manure and urine, resulting in the high level of organoarsenic compounds in the surface soils near swine farms [2,3]. Although the toxicity of organic arsenic is less than that of inorganic arsenic, long-term usage could inhibit activity of enzyme, and lead to the collapse of animal tissues and organs [4]. Meanwhile, the high solubility of ROX in aqueous solution resulted in its high leakage risk from manures, which might lead to water pollution by organic arsenic [5]. Moreover, these organoarsenic compounds introduced into the natural environment via animal manure and urine could be converted into dimethylarsinic...
acid (DMA) and inorganic arsenic (iAs) with much higher toxicity and mobility through biotic or abiotic processes [2,6–9]. Therefore, understanding the migration and transformation behavior of organoarsenic compounds in natural environment is of great significance.

The previous studies revealed that metal (oxyhydr) oxides, especially iron (oxyhydro)oxides, an important component in soil and sediments, played a vital role in adsorption of plentiful arsenic species, including inorganic and organic arsenic [10–15]. For example, Xie and Cheng [10] revealed that arsenic acid group of p-ASA and ROX could form inner-sphere complexes on the surface of iron (oxyhydro)oxides and γ-MnO₂. And those organoarsenic compounds adsorption processes were promoted by Fe³⁺ capable to form hydrous ferric oxide precipitation with high affinity to arsenicals, but barely affected by co-existed cations (i.e., Ca²⁺, Cu²⁺, Mg²⁺, and Zn²⁺). Zhang et al. [16] revealed that molecular configuration of organic arsenics adsorbed on hematite depended on exposed facet of hematite. Chen et al. [17] found that the similar adsorption trends of ROX and p-ASA with goethite and aluminum oxide could be found, while the surface adsorption site of ALO₂⁻ was approximately 3 times lower than that for FeOOH. Based on these studies, iron (hydro)oxide was a kind of reactive minerals which could adsorb organic arsenics. Considering that iron (oxyhydro)oxides often coexisted with divalent manganese ions (Mn(II)) originated from the reduction of Mn mineral in many redox-active environments, and iron (oxyhydro)oxides could catalyze the oxidation of Mn(II) by oxygen to generate minerals with Mn(III), including MnO₂₅₋, β-MnOOH, and γ-MnOOH, which could oxidize organic matter [18–21]. It was speculated that adsorption and oxidation might be involved during the organic arsenic removal process by iron (oxyhydro)oxides in the presence of Mn(II).

To better understand the interaction among (oxyhydro) oxides, organoarsenic, and Mn(II) in environment, hematite and ROX were selected as model (oxyhydro)oxides and organic arsenics [2,6–9]. Therefore, understanding the migration and transformation behavior of organoarsenic compounds in natural environment is of great significance.

The specific XRD-7000X (Shimadzu, Japan) was used to determine the component and purity of hematite and Mn(II) used throughout the experiments. The concentration of ROX was analyzed by a high-performance liquid chromatography (HPLC, 1260 Infinity, Agilent) equipped with an Agilent reversed-phase column (ZORBAX SB-C18, 150 mm × 4.6 mm, 5 µm). The mobile phase consists of 70% phosphoric acid (0.05 mmol L⁻¹) and formic acid (0.1%), 30% methanol at the flow rate of 1.0 mL min⁻¹. The absorption wavelength of UV detector was fixed at 260 nm. In order to determine the amount of adsorbed ROX on the surface of Fe₂O₃ with the addition of Mn(II) and residual ROX in solution during the removal process, concentrated hydrochloric acid was used to dissolve Fe₂O₃ powder, and the final solution was analyzed by HPLC. For the detail procedure, 5 mg Fe₂O₃ particles and 0.05 mmol L⁻¹ Mn(II) was added into 5 mL ROX (20 mg L⁻¹) solution. Subsequently, 5 mL of 66.7% hydrochloric acid was added into the suspension solution containing Fe₂O₃ particles and ROX. To accelerate the dissolution process, the suspension was stirred with fixed rate about 1 min at 70°C. And then, the dissolved suspension solution was filtered using 0.22 µm PTFE membrane, and the ROX concentration was analyzed by HPLC. Formaldoxime colorimetric method and electron spin resonance spectrum (ESR) were used to measure the concentration of dissolved Mn(II) [22]. As the initial Mn(II) concentration in reaction solution exceed detection limit by formaldoxime colorimetric method, samples were diluted by 100 times before tested.

2.4. Characterization

The morphology and crystal structures of Fe₂O₃ before and after reaction were recorded by scanning electron microscopy (SEM, SU8010) and X-ray diffraction (XRD, Shimadzu XRD-7000X) equipped with Cu Kα radiation. The specific surface area of Fe₂O₃ was analyzed via N₂ adsorption and desorption curves (Micromeritics 2460). The elements valence on the surface of Fe₂O₃ were measured by XPS (Thermo Scientific K-Alpha). The zeta potential of hematite in the absence or presence of Mn(II) with pH values ranged from 3 to 10 was determined by DTS 1070 Zetasizer and nanoparticle size analyzer (Malvern Panalytical, Malvern, UK) using 10 mmol L⁻¹ NaCl as the background electrolyte. ATR-FITR spectra were collected by FTIR spectrometer (Nicolet 6700, Thermo) equipped with a diamond internal reflection unit and a mercury-cadmium-telluride (MCT) detector. The detail analysis information was the same as literatures [16].

2.1. Chemicals and materials

ROX (4-hydroxy-3-nitrophenoxyarsonic, >98.0%) was purchased from TCI Chemicals (Shanghai, China), Fe₂O₃ (99.7%) was obtained from Aladdin Reagent (Shanghai) Co., Ltd., China without further purification, manganese(II) chloride tetrahydrate, sodium hydroxide, and other reagents were of analytical grade and provided by Sinopharm Chemical Reagent Co., Ltd., China. All reagents were used as received without further treatment. Deionized water was used throughout the experiments.

2.2. ROX removal with hematite and Mn(II)

In a typical process, 0.05 g of Fe₂O₃ powder was added into a 100 mL conical flask containing 50 mL of ROX (20 mg L⁻¹) solution in the presence or absence of 0.5 mmol L⁻¹ Mn(II) without pH adjustment unless specified statement, and 10 mmol L⁻¹ NaCl solution was used as the background electrolyte. The reaction vessel was placed on a gyrotorn oscillator open to air at 25°C with fixed oscillation rate of 220 rpm, and samples were collected at regular time interval by syringe-filtered using 0.22 µm polytetrafluoroethylene (PTFE) membrane for the subsequent ROX concentration analysis.

2.3. Analytical methods

The concentration of ROX was analyzed by high-performance liquid chromatography (HPLC, 1260 Infinity, Agilent) equipped with an Agilent reversed-phase column (ZORBAX SB-C18, 150 mm × 4.6 mm, 5 µm). The mobile phase consists of 70% phosphoric acid (0.05 mmol L⁻¹) and formic acid (0.1%), 30% methanol at the flow rate of 1.0 mL min⁻¹. The absorption wavelength of UV detector was fixed at 260 nm. In order to determine the amount of adsorbed ROX on the surface of Fe₂O₃ with the addition of Mn(II) and residual ROX in solution during the removal process, concentrated hydrochloric acid was used to dissolve Fe₂O₃ powder, and the final solution was analyzed by HPLC. For the detail procedure, 5 mg Fe₂O₃ particles and 0.05 mmol L⁻¹ Mn(II) was added into 5 mL ROX (20 mg L⁻¹) solution. Subsequently, 5 mL of 66.7% hydrochloric acid was added into the suspension solution containing Fe₂O₃ particles and ROX. To accelerate the dissolution process, the suspension was stirred with fixed rate about 1 min at 70°C. And then, the dissolved suspension solution was filtered using 0.22 µm PTFE membrane, and the ROX concentration was analyzed by HPLC. Formaldoxime colorimetric method and electron spin resonance spectrum (ESR) were used to measure the concentration of dissolved Mn(II) [22]. As the initial Mn(II) concentration in reaction solution exceed detection limit by formaldoxime colorimetric method, samples were diluted by 100 times before tested.

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3. Results and discussion

3.1. Removal of ROX by Fe$_2$O$_3$ in the presence and absence of Mn(II)

To evaluate the effect of Mn(II) on ROX removal by Fe$_2$O$_3$, we monitored the ROX concentration variation with or without the addition of Mn(II). As presented in Fig. 1a, the percentages of ROX removal with Fe$_2$O$_3$ within 96 h in the absence and presence of Mn(II) were 67.16% and 88.38%, respectively, suggesting that Mn(II) enhanced the ROX removal efficiency significantly. As the control, Mn(II) alone could not remove any ROX. In addition, the ROX removal curves with or without the addition of Mn(II) fitted well with the pseudo-second-order non-linear kinetic model of high correlation coefficients ($R^2 > 0.999$) (Fig. 1b). Considering that the specific surface area of Fe$_2$O$_3$ was 131 m$^2$ g$^{-1}$, the apparent ROX removal rate constant of Fe$_2$O$_3$ with the addition of Mn(II) was calculated to be $4.5 \times 10^{-3}$ g m$^{-2}$ h$^{-1}$, which was 4.8 times than that without the addition of Mn(II). Moreover, to further confirm the kinetic model for ROX removal process, zero-order and pseudo-first-order kinetics fitting were further analyzed (Figs. 1c and d), and the related parameters of coefficient of determination ($R^2$), chi-square, and sum of the squares of the errors (SSE) are summarized in Table 1. It was found that the values of $R^2$ for zero-order and pseudo-first-order kinetic fitting curves in the presence and absence of Mn(II) were only 0.692, 0.736, 0.570, and 0.767, respectively, suggesting that the zero-order and pseudo-first-order kinetic equations were not suitable for the ROX removal process. Meanwhile, values for the squares of errors and the chi-square test with the pseudo-second-order kinetic fitting were reasonable [23]. Therefore, it could be concluded that the ROX removal process obeyed pseudo-second-order kinetic model.

Subsequently, to determine the maximum immobilization capacity of Fe$_2$O$_3$ with or without the addition of Mn(II), the sorption isotherms based on the different initial ROX concentrations ranged from 20 to 250 mg L$^{-1}$ were investigated. It was found that the presence of Mn(II) indeed promoted the ROX removal percentage, and the ROX removal percentage decreased from 88.46% to 30.80% with the increase of ROX concentration (Figs. 2a and b). Moreover, Langmuir isotherm model and Freundlich isotherm model were utilized to analyze the ROX adsorption equilibrium. The linear formula of Langmuir equation could be written as Eq. (1) [17].

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad (1)$$

where $C_e$ (mg L$^{-1}$) is the equilibrium concentration of ROX, $q_e$ (mg g$^{-1}$) is the ROX equilibrium adsorption amount, $q_m$ (mg g$^{-1}$) and $K_L$ (L mg$^{-1}$) were the maximum adsorption capacity and the equilibrium constant, respectively.

![Fig. 1.](image-url) (a) Time profiles of ROX removal with Fe$_2$O$_3$ in the presence and absence of Mn(II); (b) pseudo-second-order, (c) zero-order, and (d) pseudo-second-order kinetics fitting curves of ROX removal in the Fe$_2$O$_3$ and Fe$_2$O$_3$/Mn(II) system. The initial concentrations of Fe$_2$O$_3$, Mn(II) and ROX were 1.0 g L$^{-1}$, 0.5 mmol L$^{-1}$ and 20.0 mg L$^{-1}$, respectively. 10 mmol L$^{-1}$ NaCl was used as the background electrolyte. Error bars represent ± one standard deviation derived from triplicate experiments.
The linear form of the Freundlich model could be represented as Eq. (2) [24].

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e$$

(2)

where $K_f$ (mg$^{-1}$ g$^{-1}$ L$^{-n}$) was attributed to adsorption capacity, and $1/n$ was Freundlich constants related to adsorption intensity. The related equilibrium adsorption isotherm fitting parameters are summarized in Table 2. We found that ROX removal process fitted better with the Langmuir model than the Freundlich model (Figs. 2c and d), indicating that monolayer coverage was preferred for ROX adsorption on Fe$_2$O$_3$.

### 3.2. Oxidation and adsorption ratio of removed ROX

According to the previous studies, Mn(II) adsorbed on Fe$_2$O$_3$ surface could be oxidized by molecular oxygen (O$_2$)
to form Mn(III, IV) oxides and oxyhydroxides [19,21], and the formed Mn minerals could oxidize the organic matter in turn. Therefore, the enhanced ROX removal efficiency with the addition of Mn(II) might be attributed to the promoted ROX oxidation process by the manganese minerals in this study. Meanwhile, as the toxicity of inorganic arsenic was much higher than that of organic arsenic compounds, and the oxidation of ROX usually resulted in the cleavage of C-As bond, leading to the generation of inorganic arsenic, such as arsenite and arsenate [14], therefore, it is of great significance to reveal the ratio of oxidized ROX to adsorbed ROX in the presence and absence of Mn(II). Since molecular oxygen was involved in the Mn(II) oxidation process, the ROX removal rate would be decreased when dissolved oxygen (DO) was eliminated from aqueous solution if both oxidation and adsorption processes involved in the Fe$_2$O$_3$/Mn(II) system. Therefore, we compared the ROX removal performance at Air and Ar atmosphere. As presented in Fig. 3a, there was no significant difference between the Fe$_2$O$_3$/Mn(II)/Air and Fe$_2$O$_3$/Mn(II)/Ar system in ROX removal curves, indicating that the oxidation mechanism was excluded during the ROX removal process in the Fe$_2$O$_3$/Mn(II)/Air system. In addition, as the oxidation degradation of ROX would result in the decrease of ROX concentration in aqueous solution and on Fe$_2$O$_3$ surface, we therefore measured unreacted ROX in aqueous solution and ROX adsorbed on the iron oxide surface by dissolving the Fe$_2$O$_3$ with concentrated hydrochloric acid, and then analyzed the dissolved ROX concentration using HPLC. As shown in Fig. 3b, the total ROX concentration kept stable around 20 mg L$^{-1}$, further suggesting that ROX could not be degraded in the Fe$_2$O$_3$/Mn(II)/Air system.

Meanwhile, as organic matter oxidation process was caused by the Mn(II) oxidation products adsorbed on Fe$_2$O$_3$ surface, and the Mn(II) oxidation process was accompanied with the decreased Mn(II) concentration in solution, we further monitored the Mn(II) concentration variation in the Fe$_2$O$_3$/Mn(II)/Air system by formaldoxime colorimetric method and electron spin resonance spectrum (ESR). It was found that Mn(II) concentration after diluted by 100 times kept stable around 0.34 mg L$^{-1}$ (Fig. 4a), indicating that neither adsorption of Mn(II) on Fe$_2$O$_3$ surface nor the oxidation of Mn(II) catalyzed by Fe$_2$O$_3$ could occur in this study. This conclusion could be further confirmed by ESR results as the signal intensity attributed to Mn(II) did not decreased with time (Fig. 4b). To further verify that Mn(II) did not transform into Mn(III), pyrophosphate (PP) was added into the Fe$_2$O$_3$/Mn(II) system since PP could form complex with Mn(III), which could be monitored by UV-Vis spectra [25]. However, no apparent peak around 258 nm assigned to the complex of PP and Mn(III) was found, indicating that Fe$_2$O$_3$ could not oxidize Mn(II) into Mn(III) (Fig. 4c).

3.3. Characterizations of Fe$_2$O$_3$ before and after ROX removal reaction

To determine whether the morphologies and crystalline structures of Fe$_2$O$_3$ varied after ROX removal, SEM images and XRD patterns were collected. As presented in Figs. 5a and b, Fe$_2$O$_3$ before and after ROX removal with

Table 2

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<td>$R^2$</td>
<td>$K_F$ (mg$^{1-n}$ g$^{-1}$ L$^{-n}$)</td>
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<td>13.96</td>
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<td>Fe$_2$O$_3$</td>
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Fig. 3. (a) Effect of solution dissolved oxygen on ROX adsorption by Fe$_2$O$_3$ and (b) concentrations of ROX absorbed on Fe$_2$O$_3$ surface and residual in solution vs. time. The initial concentrations of Fe$_2$O$_3$, Mn(II) and ROX were 1.0 g L$^{-1}$, 2.0 mmol L$^{-1}$ and 20.0 mg L$^{-1}$, respectively. Error bars represent ± one standard deviation derived from triplicate experiments.
the addition of Mn(II) showed rod-like structure around 200–500 nm in length. Peaks of samples before and after reaction matched well with the JCPDF file No. 99-0060 (Fig. 5c). Subsequently, to further confirm that Mn(II) could not be oxidized by Fe$_3$O$_4$ or be adsorbed on the surface of Fe$_3$O$_4$, we used XPS to characterize the surface elements composition and valence of Fe$_3$O$_4$ before and after reaction. It was found that there was no peaks attributed to Mn element in the full-range and high-resolution XPS spectra of Fe$_3$O$_4$ with the addition of Mn(II) (Figs. 6a and b), suggesting that the oxidation and adsorption process of Mn(II) could not occur on the surface of Fe$_3$O$_4$. In contrast, binding energy around 45.2 eV corresponding to As 3d was found on surface of Fe$_3$O$_4$ (Fig. 6c) with or without the addition of Mn(II), further indicating the adsorption of ROX on Fe$_3$O$_4$ [26].

According to the present studies, organic arsenics compounds adsorbed on iron oxide with different exposed facet could form different molecular configurations, resulting in different $p$-ASA adsorption efficiency [16]. To determine whether the different ROX adsorption efficiency of Fe$_3$O$_4$ with the addition of Mn(II) was caused by the different ROX adsorption configurations, representative ATR-FTIR spectra of ROX were monitored in different ROX concentrations. As shown in Fig. 7a, it was found that peaks around 1,325

Fig. 4. (a) Mn(II) concentration variation in the presence of Fe$_3$O$_4$ and ROX monitored by UV-Vis spectrum and (b) Mn(II) concentration variation monitored by electron spin resonance spectrum; (c) UV-Vis spectrum reaction solution in the Fe$_3$O$_4$/Mn(II) system with the addition of pyrophosphate (PP). The initial concentrations of Fe$_3$O$_4$, Mn(II), ROX, and PP were 1.0 g L$^{-1}$, 2.0 mmol L$^{-1}$, 20.0 mg L$^{-1}$ and 50 mmol L$^{-1}$, respectively. Error bars represent ± one standard deviation derived from triplicate experiments.

Fig. 5. SEM images of (a) Fe$_3$O$_4$ before reaction, (b) Fe$_3$O$_4$ after reaction in the presence of Mn(II). (c) XRD patterns of Fe$_3$O$_4$ before and after reaction in the presence of Mn(II).

Fig. 6. (a) Full-range XPS spectra of Fe$_3$O$_4$ powder in the presence and absence of Mn(II) before and after ROX adsorption. High-resolution XPS spectra of Fe$_3$O$_4$ powder before and after ROX adsorption for (b) Mn 2p, and (c) As 3d.
1,531 cm\(^{-1}\) were ascribed to symmetrical and asymmetrical stretching vibration of \(-\text{NO}_2\) respectively [27]. Meanwhile, peaks associated with the vibration of C\(-\text{N}\) and C\(-\text{As}\) arose at 1,260 and 1,160 cm\(^{-1}\) [28]. Moreover, the typical As\(-\text{O}\) bond originated from the \(-\text{AsO(OH)}_2\) of ROX were located at 912, 894, and 756 cm\(^{-1}\) [24]. Subsequently, we compared the ATR-FTIR spectra of ROX using Fe\(_2\)O\(_3\) as adsorbent with or without the addition of Mn(II) (Figs. 7b and c). It was found that the addition of divalent manganese ions did not affect the ROX adsorption configuration since ATR-FTIR spectra of the ROX adsorption by Fe\(_2\)O\(_3\) did not change significantly with the addition of Mn(II). Component observed at 897 cm\(^{-1}\) indicated the presence of \(-\text{AsO(OH)}_2\), and intense peak at 832 cm\(^{-1}\) could be attributed to H-bonded complex [29]. Meanwhile, component <800 cm\(^{-1}\) was assigned to inner-sphere complex As\(-\text{O}\)–Fe [29]. Based on the ATR-FTIR spectra results, it could be concluded that both H-bond complexes and inner-sphere complexes could be formed during the ROX adsorption process using Fe\(_2\)O\(_3\) as adsorbents.

3.5. Cyclic stability of ROX removal by iron(III) oxide and Mn(II)

Based on the above experiment results, it could be concluded that Mn(II) improved the ROX adsorption process by Fe\(_2\)O\(_3\). To further confirm this conclusion, 0.1 mol L\(^{-1}\) NaOH aqueous solution was utilized to elute the adsorbed ROX on the surface of Fe\(_2\)O\(_3\), and then repeated the ROX adsorption experiments with the addition of Mn(II). The results showed that after five runs there was no significant decrease in ROX removal efficiency (Fig. 9), further suggesting that adsorption process was the dominant removal mechanism for ROX.

3.6. ROX removal by iron(III) oxide in real wastewater samples

To investigate the ROX removal efficiency by Fe\(_2\)O\(_3\) with or without Mn(II) in real wastewater samples, ROX was added into the swine wastewater obtained from Wuhan Xinnongyuan Farm. As shown in Fig. 10, the removal percentage of ROX with Fe\(_2\)O\(_3\) within 300 min in the presence of Mn(II) was 91.96%, which was 6.23 times that in the absence of Mn(II) (14.77%). The results show that Fe\(_2\)O\(_3\)/Mn(II) system was also applicable for ROX removal in aquaculture wastewater.

4. Conclusions

In this study, the mechanism of ROX removal by Fe\(_2\)O\(_3\) with or without the addition of Mn(II) was reported.
Mn(II) could not be oxidized to Mn(III) by O$_2$ in the presence of Fe$_2$O$_3$, and ROX could not be oxidized. Mn(II) promoted ROX adsorption percentage from 67.16% and 88.38% within 96 h via enhancing the zeta potential of Fe$_2$O$_3$ from 5.3 to 7.5, which favored organoarsenic anions removal. These findings provide us better understanding for the migration and transformation of ROX.

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