



An approach to minimizing the uncertainty caused by sediment washing pretreatment in phosphorus adsorption experiments



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ABSTRACT

The presence of native adsorptive substances (NAS) cause uncertainties in the analysis of sediment phosphorus (P) adsorptive capacity. Therefore, HCl washing pretreatment is now used to clean sediment NAS. However, uncertainties still exist because the concentration (C) varies widely, from pH 3.5–12 mol/L, among studies. In this study, sediment samples from four sites were washed with HCl at different concentrations (C), and then analyzed to evaluate washing effects on the native adsorptive substances (phosphorus, metals, and organic matter), the phosphorus equilibrium adsorption capacity (EAC), and sediment skeleton erosion. Study results reveal that washing efficiency, sediment skeleton erosion, and adsorption capacity varied with C . Micromorphology analysis showed that sediment erosion increased with increasing C ; the optimum C value is 0.0–1.0 mol/L (STG1) level based on its small negative impact on the sediment skeleton. Additionally, NAS analysis further showed that washing efficiency increased with increasing C . Most native adsorptive substances (NAP, Fe, Al, Ca, and OM) were released when the HCl concentration was in the range of 0.5–1.0 mol/L. Therefore, 0.5–1.0 mol/L (in STG1) may be the best choice for sediment washing because of its fairly satisfactory washing efficiency. Finally, P equilibrium adsorption capacity (EAC) analysis showed that the sorption capacity varied greatly with C at 0.5–1.0 mol/L, due to the erosion of the sediment skeleton. Therefore, C at 0.5 mol/L is the optimal concentration because the skeleton erosion was minimized when C decreased to the minimum point in the 0.5–1.0 mol/L range. The HCl washing pretreatment ($C = 0.5$ mol/L) can also be used for analyzing other basic physicochemical substances (P, Ca, OM, Fe, Al). Our findings are an important step in providing important data for ecological restoration of wetlands, streams, rivers, lakes, and reservoirs, especially for those controlled by the internal source.

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1. Introduction

Eutrophication is a leading cause of impairment in many freshwater and coastal marine ecosystems worldwide (Smith and Schindler, 2009). Therefore, it is a key water quality issue (Downing, 2014; Lürling et al., 2016). Numerous efforts have been made to reduce the external P load; however, the importance of an internal load released from sediment, which may persist long after the reduction/elimination of an external P load has also been identified (6 years, Köhler et al., 2005; 10 years, Søndergaard et al., 2003; 10 years, Welch and Cooke, 2005). Many mechanistic surface complexation models have been used to estimate the phosphorus

sediment–water exchange and assess the environment impacts of the internal load (Huang et al., 2015; El Nemr et al., 2016; Fang et al., 2016). In order to understand the interaction mechanism between P and sediment, a variety of experiments (Zhang et al., 2012; Li et al., 2015; Fang et al., 2014; Carneiro et al., 2016; Liu et al., 2016) have been conducted worldwide, but the generality of their results is still questionable (House and Denison, 2000; Aimin et al., 2005; Komatsu et al., 2006). One key factor is that natural sediments, even with the same combination of minerals and morphology, contain various and unpredictable native adsorbed substances (NAS) (House and Denison, 2000), including native adsorptive phosphorus (NAP), organic matter (OM), and metal salts (especially Fe, Al, and Ca). They influence the P adsorption capacity of the sediment (Ying et al., 2009; Huser and Pilgrim, 2014) and decrease the credibility of experiments (Pilgrim et al., 2007; Meis et al., 2013). For example, reducing Fe–P complexes will result in phosphate release when redox potential decreases, and P co-precipitates with low

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Table 1
Physical and chemical characteristics of sediment samples.

	OM (%)	TP (mg/kg)	TN	Fe (%)	Al (%)	Ca (%)	Clay (%)	Silt (%)	Sand (%)
Wuhan	5.22	632.47	146.17	2.77	4.19	3.93	0	34.54	65.46
Cuntan	7.26	924.77	227.45	3.00	3.97	4.81	0.94	72.78	26.28
Zhengzhou	1.42	1412.25	44.30	4.16	3.83	3.03	0	23.12	76.88
Lanzhou	4.76	549.13	153.34	1.84	3.31	6.99	2.28	47.31	50.41

solubility calcite at pH higher than 9 (Cooke et al., 2005). Smolders et al. (2006) indicated that oxygen consumption due to bacterial breakdown of organic matter may decrease the P-binding capacity of sediments due to FeS_x formation. Berg et al. (2004) suggested that several calcite characteristics, such as grain size, specific surface area, and structure determine the P adsorption capacity. These unpredictable and uncontrollable substances cause uncertainty in experiment results (Aminot and Andrieux, 1996; James, 2011; Meis et al., 2013; Pilgrim et al., 2007), as different conditions can have pronounced effects on the reported P-adsorption value. Furthermore, these effects may be site and material specific (Lüring et al., 2016).

The HCl washing pretreatment is now considered the most promising technique for cleaning NAP (Aaron et al., 2000; Zhang et al., 2012; Fang et al., 2014). However, the HCl concentration (C) used in the washing pretreatment has varied in previous studies (pH 3.5, Aaron et al., 2000; 0.5 mol/L, Snape et al., 2004; 1 mol/L, Sutherland, 2002; 12 mol/L, Fang et al., 2014; 12 mol/L, Zhang et al., 2012). This variation has a considerable impact on the sediment adsorption/desorption properties (Peng, 2007; Gao, 2012). Peng (2007) showed that the binding capability of each phosphorus fraction differed greatly under different pH conditions. Iron–phosphorus (Fe–P) and aluminum–phosphorus (Al–P) possessed the greatest binding capability at pH 7–8, and organic phosphorus extracted at alkaline conditions (OPalk) and calcium–phosphorus (Ca–P) showed the greatest binding capability at pH 5 and pH 9, respectively. This suggests that NAS will vary greatly after washing pretreatment with different C. Aimin et al. (2005) showed that NAP has a negative influence on the phosphorus equilibrium adsorption capacity (EAC) because NAP may compete for the absorptive sites. Chuai et al. (2014) reported a positive association between EAC and Ca, and EAC and OM, but a negative relationship was evidenced between EAC and NAP (Aimin et al., 2005). This indicated that experiment results were uncertain when C was not unified in the washing pretreatment. Therefore, a unified C for all the washing pretreatments should be determined. In addition, although previous studies have shown that dilute HCl, 0.5 mol/L (Sutherland, 2002) and 1.0 mol/L (Snape et al., 2004), had little negative effect on the extraction of natural geogenic metals, some studies (Ostrom et al., 1961; Yokoo et al., 2004) found that sediment silicates were destroyed when thick hydrochloric acids were used. Ostrom (1961) also found that strong concentrations (e.g., 3 mol/L) could dissolve residual sediment phases, especially for less-resistant layered aluminum-silicates. This indicates that, although C increase is beneficial for washing efficiency, it results in a negative effect on the original sediment silicates. Therefore, the choice of C is not simple when both washing efficiency and the sediment erosion skeleton are considered. Comprehensive research should be undertaken to minimize the uncertainty caused by washing pretreatment, in which both washing efficiency and erosion of sediment silicates are considered in deciding a satisfactory C.

The objective of this study was to investigate the effects of C on 1) the cleaning efficiency of adsorptive contaminants (NAP, OM, Fe, Al, and Ca); 2) erosion of the sediment skeleton, characterized by micro-morphology and surface characteristics; 3) EAC, which can help identify the uncertainty caused by C variability and washing efficiency. Finally, a satisfactory C for the washing pre-

treatment is presented based on a comprehensive analysis of these effects.

2. Methods and materials

2.1. Sediment collection and analysis

Surface sediment samples (0–15 cm) were collected using a grab sampler at four sampling stations (Cuntan, Wuhan, Lanzhou and Zhengzhou). Cuntan and Wuhan are located in the Yangtze River, the longest and largest river in China. The former is located in the upper reach and the latter in the lower reach of the Three Gorges Dam. Lanzhou and Zhengzhou are located in the upper and lower reaches, respectively, of the Yellow River, the second-longest river in China. Samples were immediately transported to the laboratory in sealed plastic bags. After removing coarse plant fragments, a portion of the samples were freeze-dried (2–4 LSC; Christ, Germany) for analysis of physicochemical properties and the remainder were desiccated at room temperature. The sediment was then homogenized and sieved into two size fractions: D1 (<0.05 mm) and D2 (0.1–0.2 mm). For the Cuntan sample, where the maximum grain size was about 0.1 mm, the coarse sediment particle size was D3 (0.05–0.1 mm). All samples were stored in plastic packs for further analysis.

The sediment elemental compositions of Al, Fe, Ca, and Si were measured with an X-ray fluorescence spectrometer (XRF-1800; Shimadzu, Japan). The grain size fractions were measured with a Laser grain size analyzer (S3500; Microtrac, USA) and the sediments were further separated into clay (<0.002 mm), silt (0.002–0.05 mm) and sand fractions (0.05–2 mm) (Jin et al., 2005). The organic matter (OM) was analyzed using the combustion method at 550 °C. After digestion of the samples with H₂SO₄ and HCl, total nitrogen (TN) and total phosphorus (TP) were measured by colorimetry. The basic substance/element content of sediment samples are presented in Table 1.

2.2. Sediment washing experiment

The acid washing process was carried out in a glass conical flask with a plug under atmospheric pressure at 25 °C for 2 h as follows: 1) HCl at specific concentrations (C = 0, 0.1, 0.5, 1, 3, 5, 8 and 12 mol/L) and sediment samples were loaded into glass reactors with a liquid to solid ratio of 10 mL/g, and placed into a constant temperature oscillator at 160 r/min at staggered intervals; 2) sub-samples were then filtered under vacuum, washed with distilled water until the filtrate was neutral and dried at 110 °C to constant weight. The sediment was stirred and agitated constantly to mitigate sediment compaction during the drying process. Sub-samples were labeled S0, S0.1, S0.5, etc., according to C and were separately put in sealed plastic bags and stored in a desiccator for further analysis.

2.3. Micromorphology and surface characteristics

The micromorphology of the HCl-washed sediment was investigated with a scanning electron microscopy (SEM). The dried samples were glued on an aluminum sample holder using conductive silver tape and sputter-coated with 20 nm gold (JFC 1200;

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