



# Effect of extraction conditions on citrate-mediated phosphorus removal and recovery from intercepted particulate runoff

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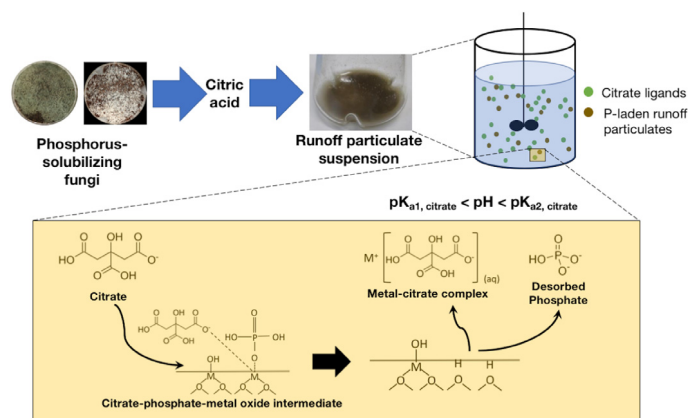
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## HIGHLIGHTS

- Runoff P release was favored at low pH and sediment loading and high citrate dose.
- Ligand-induced P release appeared optimal at pH between citrate  $pK_{a1}$  and  $pK_{a2}$ .
- Fed-batch or semi-continuous extraction with pH control could improve P extraction.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Phosphorus (P) is a nonrenewable mineral resource that is critical to achieving food security for the growing world population. However, nearly half of total annual mined P is lost through nonpoint particulate runoff streams. Previously, we demonstrated inorganic phosphate extraction from runoff particulate mineral phases using P-solubilizing fungi (PSF) generating the organic acids citrate and oxalate. The objective of the current study was to investigate the effect of extraction process conditions on P extraction efficiency and kinetics from impounded urban storm water runoff sediments using citrate as the sole P solubilization agent. Abiotic extraction experiments were conducted according to a one-factor-at-a-time approach. High P removal and solubilization were favored at low sediment loading (5 g/100 mL), low initial pH (3), and high initial organic acid loading (75 mM), with P recovery efficiencies of 75% to 100% and 240 ppm solubilized phosphate levels achieved under these conditions. Initial and equilibrium extractant pH levels between citrate  $pK_{a1}$  (3.08) and  $pK_{a2}$  (4.74) appeared conducive for citrate ligand-induced particulate P release,

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possibly due to optimal balance of protonated and dissociated carboxylic groups. The results suggest that operating the extraction under fed-batch or semi-continuous modes with pH control can help maximize runoff particulate P recovery efficiency in PSF-mediated bioextractions wherein citric acid is the major fungal organic acid product.

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## 1. Introduction

Modern agriculture relies on non-renewable phosphorus (P) ores to sustain food crop production at commercial quantities to meet the needs of the continuously growing world population. While global phosphate rock quantity and quality are declining and becoming more concentrated in limited geopolitical regions in the world (Smit et al., 2009), massive environmental P losses occur annually through point and nonpoint pollution discharges (Cordell et al., 2009). Majority of current P remediation and reclamation efforts focus on soluble reactive phosphates in point sources such as domestic and agricultural wastewaters while particulate-bound P in nonpoint runoff and erosion streams are largely ignored (Rittmann et al., 2011). These nonpoint P losses account for 46% of total annual mined P (Cordell et al., 2009). Recovering these losses is considered a significant step in closing the disjointed global P loop, which leads to pollutant nutrient overloads in natural water resources around the globe (NSF, 2014). Therefore, achieving total P recovery by addressing nonpoint losses will not only help improve water resources quality but also provide societal benefits by helping ensure food resource security (Mayer et al., 2016).

P-solubilizing fungi (PSF) can mobilize mineral-bound phosphate ions (inorganic P) and ester-bound phosphate in organic P compounds (i.e., organic P fraction) by generating and using chelating and solubilizing agents (e.g., low molecular weight organic acids or LMWOAs), and phosphatase enzymes, respectively (Sharma et al., 2013). As such, PSF have been used to reduce phosphate ion fixation on soil Fe- and Al-(oxide) minerals (Osorio and Habte, 2014, 2013) and improve solubility of Ca-phosphate mineral fertilizers (Osorio Vega et al., 2015; Schneider et al., 2010); thus, enhancing soil P mobility and helping improve plant P uptake efficiency and crop biomass yield. Prior studies have attributed bound phosphate mobilization from soil minerals by LMWOAs to ligand exchange, ligand-induced dissolution, and/or proton-induced dissolution mechanisms (Bhatti et al., 1998; Fox and Comerford, 1992; Johnson and Loeppert, 2006; Lindgren and Persson, 2009; Schefe et al., 2009; Traina et al., 1986). Despite this, applying LMWOA-mediated phosphate mobilization processes to recover P from runoff particulates containing complex assemblages of similar P-binding soil Fe-, Al-, and Ca-minerals has not been attempted.

We recently published the initial results of our study, which to our knowledge is the first demonstration of the feasibility of using LMWOAs generated by PSF to extract bound P from impounded runoff particulates in a storm water sediment ditch (Mondala et al., 2017). Results showed that under abiotic conditions, aqueous pure citrate and 2:1 (mM ratio) oxalate-citrate mixture extractant solutions provided the best runoff particulate-bound P removal efficiency compared to pure oxalate and other oxalate-citrate (1:1, and 1:2 mM ratio) solutions. While 2:1 (mM) oxalate-citrate extractant mixtures produced higher reductions in the Fe-bound P (Fe-P) fraction compared to pure citrate, the use of the latter generally resulted in higher overall bound P reduction and less phosphate re-adsorption or co-precipitation with solubilized metal cations (e.g.,  $\text{Ca}^{2+}$ ) and oxalate ligands on the particulate mineral surfaces as the extraction process progressed, which likely led to the observed Ca-P fraction increases (Mondala et al., 2017). We anticipate that with pure citrate, solubilized metal cations are chelated in solution, which could minimize their interaction with precipitable ligands such as oxalate and phosphate. The current study investigated the influence of batch abiotic extraction conditions on P release kinetics and overall extraction yields from P-laden storm water runoff particulates. In this paper, we report the results of extraction studies using pure aqueous citrate solutions as the P extractant phase. These findings will be used to develop a preliminary understanding of the underlying mechanisms of P recovery in this process, which will inform engineering design and the development of scalable and field-deployable technologies that will employ this methodology.

## 2. Materials and methods

### 2.1. Runoff sediment collection and preparation

Runoff sediments used in the extraction experiments were collected from a sediment ditch of a storm water sewer drain leading to Woods Lake, a hypereutrophic isolated kettle lake system in a residential area in Kalamazoo, MI (N 42.260833°, W 85.618674°). Samples were taken from the top ~2-cm layer of the sediment mass in the ditch, washed with 1:8 w/v of DI water, air-dried, lyophilized (Labconco, Kansas City, MO, USA), and sieved through a mesh 16 sieve. A single batch of sediment samples was used in all experiments related to this study. Freeze-dried sediment samples had on average approximately 5% moisture remaining.

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