# Phosphorus source—sink relationships of stream sediments in the Rathbun Lake watershed in southern Iowa, USA

Najphak Hongthanat · John L. Kovar · Michael L. Thompson · James R. Russell · Thomas M. Isenhart

**Abstract** The surface waters of Rathbun Lake watershed in southern Iowa are impacted by agricultural sources of sediments and nutrients, including phosphorus (P). Because stream sediments often play an important role in regulating P concentrations in stream water, we investigated sediment–water column P relationships in four creeks within the watershed and then evaluated the relationship between sediment properties and indicators of the risk of P loss. Based on Mehlich-3-extractable P (17 to 68 mg kg$^{-1}$) and degree of P saturation (2 to 12 %), stream bank and bed sediments at the four sites were unlikely to serve as major sources of P. However, equilibrium P concentrations, which ranged from 0.02 to 0.12 mg L$^{-1}$, indicated that bed sediments could release P to the water column depending on dissolved P (DP) concentrations in the stream water and the time of year. The likelihood of P desorption from the sediments increased with increasing pH ($r = 0.92$, $p < 0.01$) and sand content ($r = 0.78$, $p < 0.05$), but decreased with clay content ($r = -0.72$, $p < 0.05$) and iron (Fe) ($r = -0.93$, $p < 0.001$) associated with organic matter. From these results, we speculate that changes in land use within the riparian areas may, at least initially, have little effect on P concentrations in the streams. Low concentrations of DP relative to total P (TP) in these streams, however, suggest that P loads to Rathbun Lake can be reduced if P inputs from eroded bank sediments are controlled.

**Keywords** Stream sediments · Riparian pastures · Phosphorus losses · Degree of P saturation · Equilibrium P concentration

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## Introduction

Nonpoint-source pollutants are a primary water quality problem in the USA (USEPA 2003). In Iowa, sediment has been frequently identified as an agricultural pollutant impacting water quality (IDNR 2015a). Sediment is not only detrimental to water quality, clarity, and aquatic life, but it is also a major source of P in streams (Becher et al. 2001). Because P is a primary limiting nutrient in fresh water bodies, P enrichment often leads to excessive growth of algae and other aquatic plant species (Djodjic et al. 2004). As plants die and decompose, much of the oxygen in water is consumed, resulting in a hypoxic environment and eutrophic conditions (Carpenter et al. 1998).

Sediment and P losses from agricultural systems depend on many factors, including soil characteristics, land

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use, erosion potential, and amount of runoff (Laubel et al. 2003). Jacobson et al. (2011) found that the fraction of land in row crops and the use of fertilizer P were correlated with total and dissolved P loads, respectively, in the Mississippi River basin. Research by Alexander et al. (2008), however, indicated that 37 % of the P delivered to the Gulf of Mexico originated from pasture and rangeland where manures from grazing animals were present and/or fertilizers had been applied. In Iowa, Downing and Kopaska (2000) showed that watersheds with greater portions of land in grazed pastures had higher sediment and P in surface water than did watersheds in cropland, although input pathways were not identified. More recent research in Iowa has indicated that pasturelands can be major contributors of sediment and nutrients to surface water resources if improper

grazing management reduces vegetative cover (Zaimes et al. 2004; Zaimes et al. 2008; Nellesen et al. 2011).

In the Rathbun Lake watershed in southern Iowa (Fig. 1), the Iowa Department of Natural Resources (IDNR) reported that ponds and streams are impacted by agricultural nonpoint sources of pollution, such as sediments, nutrients, and pesticides (IDNR 2015a). Livestock grazing on more than 400 privately held commercial farms in the watershed has been identified as a contributor of sediment and P to Rathbun Lake (USACE 2007). Use of streams as a water source for grazing cattle is a common practice in the watershed, and research has shown that increasing cattle stocking rates per unit of stream length will increase manure cover and decrease forage height in riparian areas (Bear et al. 2012), which may increase the risk of sediment and P losses in runoff. Tufekcioglu et al.
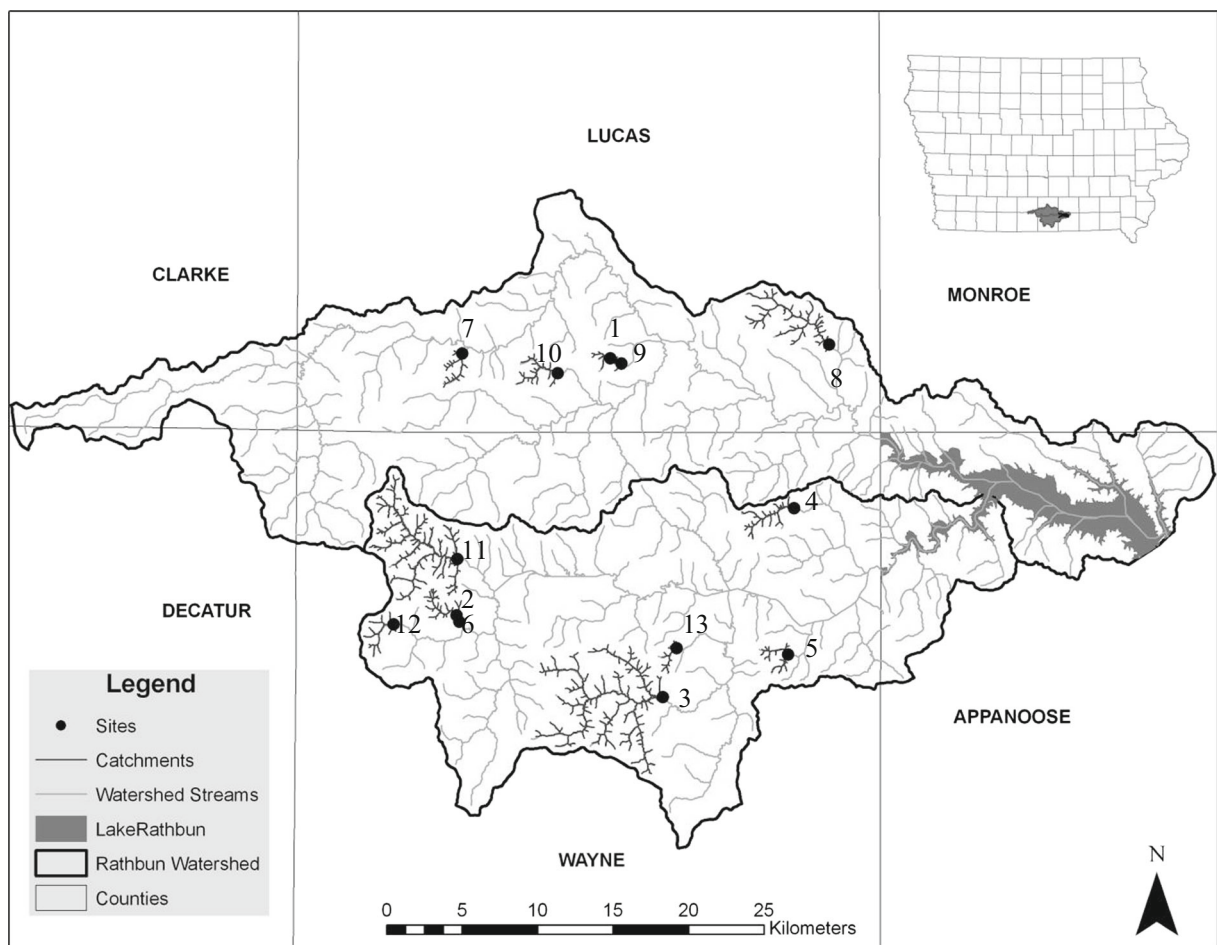


Fig. 1 Rathbun Lake watershed map showing the 13 study locations and their upstream channel systems as described by Tufekcioglu et al. (2012). Sites 8, 3, 4, and 11 are equivalent to sediment sample collection sites M, H, B, and P, respectively. The perennial streams that flow through sites M, H, B, and P are Honey Creek, West Jackson Creek, Walker Creek, and Ninemile Creek, respectively

(2012) also found that cattle stocking rates were positively correlated with the length of severely eroded stream banks and compaction of soils in riparian areas.

Several indices have been developed for evaluating the risk of P loss from upland and riparian soils. For example, the degree of P saturation (DPS) refers to the proportion of the P sorption capacity that is occupied by P (Schoumans 2009). The amounts of extractable P, Fe, and aluminum (Al) are used on a molar basis to calculate DPS. Some states, such as Delaware (Sims et al. 2002) and Florida (Nair et al. 2004), have adopted the DPS to estimate risk of P loss from agricultural soils. We recently explored the use of such estimators in assessing the risk of P loss from soils in the Rathbun Lake watershed and concluded that additional studies were needed to establish connections between these indices and water quality (Hongthanat et al. 2011).

Stream sediments play an important role in regulating soluble P concentrations in stream water, but the interaction between chemical and transport processes can be complex. The bioavailability of sediment-bound P varies with the physicochemical properties of the sediments, which reflect sediment origin and land use history (McDowell et al. 2003). The processes that control sediment transport are influenced by stream velocity and the aggregate stability of the sediments. Quite often, these processes favor transport of finer particles that have higher P sorption capacity than coarser sediments (McDaniel et al. 2009).

The concept of equilibrium phosphorus concentration (EPC) is often used to determine whether stream sediments act as a sink or a source of P for surface water (Haggard et al. 2007; McDaniel et al. 2009). In this context, the EPC is the concentration of P in solution at which there is no net adsorption or desorption of P (Taylor and Kunishi 1971). The likelihood of P release from sediments into water depends on the EPC of sediments and the amount of dissolved P (DP) in the water. If sediment EPC and water column DP are not approximately equal, the sediments and water column are not in equilibrium with respect to DP. In principle, if DP in the stream is greater than the EPC of sediments, the sediments will sorb P, and if DP is less than EPC, the sediments will release P to stream water. The higher the EPC, the more likely it is that the sediment will release P to reach equilibrium. Sediments with low EPC require lower P concentration in water to release P.

Environmental factors can affect sediment EPC. The EPC increases when sediments become anoxic (Reddy et al. 1998; Pant and Reddy 2001). House and Denison

(2000) found that the EPC of river sediments increased with decreasing redox potential. Under anaerobic conditions, crystalline forms of Fe oxides are partially dissolved or altered to poorly crystalline or amorphous forms that have greater surface area to sorb P. Although reducing conditions may increase P sorption sites, those sorption sites may also have a lower bonding energy for P than do sorption sites available under aerobic conditions (Patrick and Khalid 1974). This phenomenon may result in higher EPC under reduced conditions than under oxidized conditions (Reddy and DeLaune 2008). Haggard et al. (2004) found that the addition of alum ($Al_2(SO_4)_3$) and lime ($CaCO_3$) to sediments from four Arkansas streams decreased EPC and increased the ability of the sediments to sorb dissolved P from stream water. Other factors affecting EPC include particle size distribution (Haggard et al. 1999), ionic strength of background solution, and pH (Koski-Vähälä and Hartikainen 2001).

The prevailing concentration of P in water can also affect sediment EPC. Ekka et al. (2006) found that P discharges from a wastewater treatment plant increased background P concentrations in Ozark streams, which led to higher EPC values in stream sediments, and reduced the ability of sediments to retain P. Jarvie et al. (2005) found that bed sediments released P to water when there was large hydrological dilution of stream water after significant rainfall events.

Clearly, the role of stream sediments as a sink for P or source of P in the water column is important in understanding P dynamics in a watershed. Our objectives in this study were (i) to evaluate sediment–water column P relationships in streams within the Rathbun Lake watershed, specifically targeting four creeks, and (ii) to investigate the relationship between sediment physicochemical properties and calculated indicators of P fate to determine whether environmental risk can be predicted by these properties. We hypothesized that an analysis of stream sediments combined with stream water monitoring can provide insight into how P inputs and sediment P losses may impact water quality within the watershed. This information can be useful in targeting source areas for remediation.

## Materials and methods

### Watershed characteristics

The Rathbun Lake watershed consists of approximately 143,395 ha located within the Loess Flats and Till Plains

ecoregion of southern Iowa. The soils in the watershed formed from loess, glacial till, or alluvium (Griffith et al. 1994) and have high erodibility, root-restrictive zones near the surface, and excessive wetness, which limit use for row-crop production (Braster et al. 2001). Approximately 468 livestock operations are located in the watershed, with the majority being beef cattle (*Bos taurus* L.) operations that rely on grassland for grazing, with little or no confinement of livestock. The Rathbun Lake watershed has 61 subwatersheds ranging in size from 1049 to 6654 ha.

Within the watershed, study sites representing 13 of the 61 subwatersheds were established in 2006 as part of a larger study designed to evaluate the effect of grazing management practices on water quality (Bear et al. 2012; Tufekcioglu et al. 2012; Fig. 1). Site selection was based on landowner permission to access a site during the study and perennial flow in all pasture stream reaches. When the study of Bear et al. (2012) was initiated, surface horizons (0–20 cm) of soils at each site were sampled to determine inherent soil properties. Of interest, total P (Crosland et al. 1995) in the soils ranged from 267 to 349 mg kg$^{-1}$, which is comparable to total P (TP) measured in other Iowa watersheds with similar soil types (Zaimes et al. 2008; Nellesen et al. 2011). Additional information describing the 13 study sites is available from Bear et al. (2012) and Tufekcioglu et al. (2012).

### Water chemistry

Water chemistry data for the streams in the Rathbun Lake watershed were collected from two sources. Archival data for the years 2000 to 2009 were obtained from the Storage and Retrieval system (STORET) database maintained by the IDNR (2015b). The data in the STORET database were obtained from stream water grab samples collected and analyzed during 10 years of intermittent sampling. Fifteen sampling sites located throughout the Rathbun Lake watershed were included in the database. Additional information about the monitoring program and sampling sites is available from Balmer (2015). Selected stream water characteristics, including TP, DP, total nitrogen (TN), nitrate-N, pH, dissolved oxygen, and total suspended sediment (TSS), were recorded from the database.

In addition to the STORET data, stream water grab samples were collected and analyzed biweekly from the 13 previously described study sites (Bear et al. 2012; Tufekcioglu et al. 2012) during the period March through November 2008 and March through November 2009. None of these 13 sampling locations was included in the STORET database. Water samples were delivered to the laboratory immediately and analyzed for DP within 7 days of delivery. An unfiltered aliquot was analyzed for TP following persulfate–sulfuric acid digestion (Pote and Daniel 2009) and colorimetric determination of P (Murphy and Riley 1962). To measure DP, a second subsample was filtered through a 0.45-μm cellulose acetate membrane, and P was determined colorimetrically (Murphy and Riley 1962). All water grab samples were analyzed for DP and TP. In 2009, TSS was also measured in each sample via Standard Methods 2540 D (APHA 2005). To provide a general overview of water chemistry in the watershed, data from the STORET database and data collected from the 13 study sites were combined and summarized (Table 1).

**Table 1** Distribution of selected stream water characteristics in the Rathbun Lake watershed during 10 years of intermittent sampling (2000–2009) at sites located throughout the watershed

| Water Chemistry | Minimum | 25th Percentile | Median | 75th Percentile | Maximum |
|---|---|---|---|---|---|
| DP[a] (mg L$^{-1}$) | <0.025 | 0.04 | 0.07 | 0.11 | 2.00 |
| Total P (mg L$^{-1}$) | <0.025 | 0.15 | 0.24 | 0.37 | 2.00 |
| Nitrate-N (mg L$^{-1}$) | <0.1 | 0.3 | 0.5 | 0.9 | 2.2 |
| Total N (mg L$^{-1}$) | 0.3 | 0.7 | 0.8 | 1.6 | 6.5 |
| pH | 7.0 | 7.6 | 7.8 | 7.9 | 8.2 |
| DO[b] (mg L$^{-1}$) | 2.9 | 6.9 | 7.9 | 9.8 | 15.1 |
| TSS[c] (mg L$^{-1}$) | 3 | 290 | 390 | 499 | 3400 |

[a] Dissolved phosphorus

[b] Dissolved oxygen

[c] Total suspended sediment

## Site selection for sediment sampling

Based on the previous study of Hongthanat et al. (2011), four sites, denoted M, H, B, and P, with a range of soil properties and soil mapping units were chosen to represent the riparian sediments within the watershed (Fig. 1). The perennial streams that flow through these sites are Honey Creek, West Jackson Creek, Walker Creek, and Ninemile Creek, respectively. Honey Creek and Walker Creek are second-order streams, while West Jackson Creek and Ninemile Creek are third-order streams (Tufekcioglu et al. 2012). The uplands and riparian areas surrounding these sites are used to graze beef cattle (Bear et al. 2012). Stream bed and bank sediments were collected at each of the four selected sites in July 2009 under base-flow conditions, providing a total of eight samples for the study. Bed sediments were collected from the upper 10 cm of the stream bottom in the thalweg of the stream channel. Bank sediments were collected to a 5-cm depth from the exposed bank surface approximately 1 m above the water level.

## Sediment physicochemical properties

Subsamples of sediment were air-dried and sieved to 2-mm particle size. Air-dried samples were used to determine pH by glass electrode (soil/water, 1:1), particle size distribution by pipette method (Gee and Bauder 1986), and total carbon (TC) and TN by high-temperature dry combustion (Nelson and Sommers 1982). Total P was determined by digesting the sample in *aqua regia* (Crosland et al. 1995) and analyzing the digest colori-metrically (Murphy and Riley 1962). Phosphorus, Ca, Mg, Fe, Al, Na, and K ($P_{M3}$, $Ca_{M3}$, $Mg_{M3}$, $Fe_{M3}$, $Al_{M3}$, $Na_{M3}$, and $K_{M3}$) were extracted in Mehlich-3 solution (Mehlich 1984) and determined by inductively coupled plasma (ICP) spectrometry. Citrate–bicarbonate–dithionite–extractable Fe, Al, and Mn ($Fe_d$, $Al_d$, and $Mn_d$) were determined by atomic absorption spectros-copy (Loeppert and Inskeep 1996). Ammonium–oxa-late-extractable P, Fe, Al, and Mn ($P_{ox}$, $Fe_{ox}$, $Al_{ox}$, and $Mn_{ox}$) were determined by ICP spectrometry (Loeppert and Inskeep 1996).

The DPS (Schoumans 2009) is a measure of the pro-portion of the P sorption capacity that is occupied by P and has been used as an estimate of P release potential (Sims et al. 2002; Nair et al. 2004). Mehlich-3- and oxalate-extractable P, Fe, Al, and Ca were used to calcu-late values for DPS: DPS-ox(Fe + Al), DPS-M3(Fe +

Al), and DPS-M3(Ca) (Eqs. 1, 2, and 3). All variables in the equations have units of millimoles per kilogram.

$$DPS{-}ox(Fe + Al) = \frac{P_{ox}}{[Fe + Al_{ox}]} \times 100 \qquad (1)$$

$$DPS{-}M3(Fe + Al) = \frac{P_{M3}}{[Fe_{M3} + Al_{M3}]} \times 100 \qquad (2)$$

$$DPS{-}M3(Ca) = \frac{P_{M3}}{[Ca_{M3}]} \times 100 \qquad (3)$$

To estimate the EPC, the methods outlined by Haggard and Sharpley (2007) were followed. Wet sed-iments (equivalent to 1 g air-dried) were weighed into 50-mL centrifuge tubes and shaken in 25 mL of stream water spiked with additional amounts of P with concen-trations of 0, 0.1, 0.25, 0.5, and 1 mg P $L^{-1}$. If the ambient P concentration in water had been, for example, 0.05 mg $L^{-1}$, then the initial P concentrations were 0.05, 0.15, 0.3, 0.55, and 1.05 mg $L^{-1}$. Three drops of chlo-roform were added to each tube to inhibit microbial activity. After shaking for 24 h, the sediment suspension was centrifuged at $1200 \times g$ for 5 min, and the clear supernatant was filtered through a 0.45-μm membrane. Phosphorus concentration in the filtrate was determined colorimetrically (Murphy and Riley 1962). Phosphorus remaining in solution (C) was plotted against P sorbed on the solid phase (S), and a line was fit to the data, using linear regression (Fig. 2). At low P concentration in solution, the relationship between C and S can be described by a simple linear equation (Haggard et al. 2007). The EPC, the P equilibrium buffering capacity (PEBC), and native P sorbed on the solid phase ($S_0$) were calculated from Eq. 4:

$$S = KC{-}S_0 \qquad (4)$$

where S is P sorbed on solid phase (mg $kg^{-1}$), C is P remaining in solution after a 24-h equilibration (mg $L^{-1}$), $S_0$ is native P sorbed on solid phase (mg $kg^{-1}$), and K (the slope) is the PEBC (L $kg^{-1}$). The PEBC indicates the ability of sediments to resist a change in P concentration in solution.

To avoid the effect of drying, bed and bank sediments used to estimate the EPC were stored field moist. All results are presented on a dry weight basis. Indigenous stream water from each site was used to suspend the

**Fig. 2** Linear regression model used to estimate equilibrium P concentration (EPC) and P equilibrium buffering capacity (PEBC) in a stream bed sediment sample (site P)
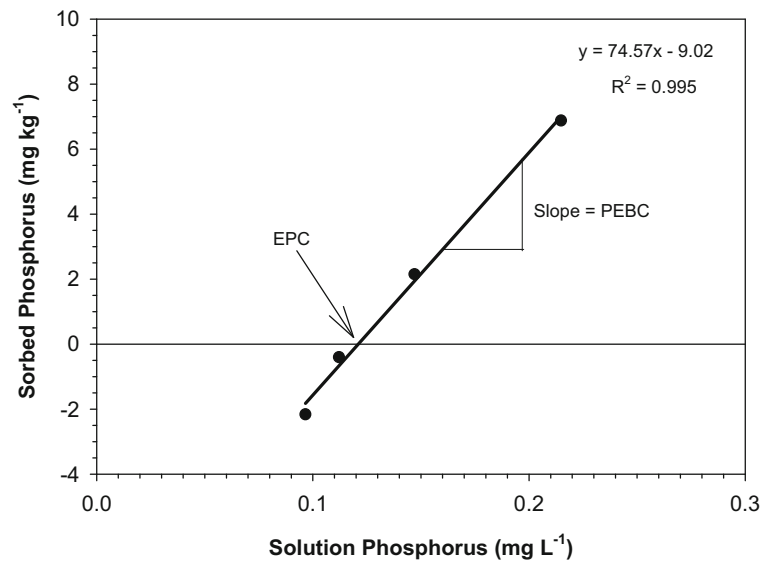


sediments. In some contexts, stream water is used as background to mimic the ionic strength ($I_e$) of the stream in which sediments occur (Haggard and Sharpley, 2007). High concentrations of dissolved cations, such as Ca, Mg, Fe, and Mn, can affect EPC measurements (House and Denison 2000), so it is useful to know the $I_e$ of the background matrix. The $I_e$ of the water at each site was estimated from the electrical conductivity (EC), using the empirical model: $I_e = 0.012$ EC (Essington 2003). The average EC of stream water was 0.4 dS m$^{-1}$; therefore, the estimated $I_e$ was 0.005 mol L$^{-1}$. Stream water samples were filtered through 0.45-μm cellulose acetate membranes and analyzed for ambient P concentration. To estimate EPC, one must begin with stream water that has an ambient P concentration lower than the EPC of sediments (Haggard and Sharpley 2007). Therefore, if the stream water initially had high P concentration, we used iron oxide-impregnated filter paper to remove P from the water. This allowed the concentrations of other solutes to be approximately maintained. When possible, we removed P from water until the P concentration was lower than the detection limit (0.005 mg L$^{-1}$) of the spectrophotometer.

Data analyses and comparisons

Statistical analyses were performed with SAS version 9.1 (SAS Institute 2003). Paired $t$ tests were used to evaluate differences in physicochemical properties between bed and bank sediments. The PROC REG procedure in SAS was used to determine correlation of sediment properties with EPC and PEBC.

Results and discussion

Surface water characteristics in the watershed

Water chemistry of the streams was typical for the Loess Flats and Till Plains ecoregion of southern Iowa (Griffith et al. 1994), with alkaline pH, generally adequate (>5 mg L$^{-1}$) dissolved oxygen (DO) for aquatic life, and relatively low nitrate-N and total N (Table 1). Nitrate-N concentrations greater than 6.0 mg L$^{-1}$ are common in surface waters in Iowa, although many watersheds have significantly more row crop production than present in the Rathbun Lake watershed (Jaynes et al. 1999; Tomer et al. 2008; Hatfield et al. 2009). Phosphorus concentrations in the streams within the watershed, however, are high, with median values of stream water DP and TP of 0.07 and 0.24 mg L$^{-1}$, respectively (Table 1). The median TP concentration greatly exceeds the proposed nutrient criteria value for wadeable, warm-water streams (0.10 mg L$^{-1}$) in Iowa (IDNR 2015c). Total P concentrations greater than 0.10 mg L$^{-1}$ may lead to eutrophication of fresh water bodies (Correll 1998). With TSS levels often exceeding 150 mg L$^{-1}$, the streams in the watershed carry significant amounts of sediment, likely a result of stream bank erosion (Zaimes et al. 2008; Tufekcioglu et al. 2012). Seasonal sediment and nutrient loads were not calculated as sufficient stream flow data are not available.

At the four sites where bank and bed sediments were collected, stream water DP concentrations in 2009 ranged from 0.065 mg L$^{-1}$ in Ninemile Creek at site P to

0.145 mg L$^{-1}$ in West Jackson Creek at site H. Total P concentrations varied little, ranging from 0.30 mg L$^{-1}$ in Ninemile Creek to 0.40 mg L$^{-1}$ in West Jackson Creek (Table 2). Dissolved P as a percentage of TP also varied little, with a low value of 22 % for Ninemile Creek (site P) up to 36 % for West Jackson Creek (site H). These percentages are smaller than those reported (Tomer et al. 2008) for the South Fork of the Iowa River (50 to 100 %), which had much lower TP concentrations (0.04 to 0.09 mg L$^{-1}$) than found in these four streams. Mean TSS concentrations were highest in Honey Creek (site M) and lowest in West Jackson Creek (site H), but variability at each site during the sampling period was significant (Table 2). Total suspended sediment concentrations at these four sites were much higher than those reported in a 10-year study by Schilling et al. (2011) for two watersheds in central Iowa in which the highest TSS concentration recorded was 167 mg L$^{-1}$. As pointed out by Tufekcioglu et al. (2012), improved riparian pasture management in the Rathbun Lake watershed could reduce sediment and P losses from stream banks and adjacent riparian areas.

Characteristics of bed and bank sediments

Physicochemical properties of the bed and bank sediments from the four selected sites varied more with sampling position (bed versus bank) than location in the watershed (Table 3). The pH values ranged from 6.5 to 8.2, with mean bed sediment pH higher than that of bank sediments. The pH values of the bank sediments were similar to those of the floodplain soils surrounding the sampling sites (Hongthanat et al. 2011). The physical composition of the bed sediments in these second- and third-order streams was coarser than that of the bank sediments, which may reflect downstream transport of finer material with stream

flow (McDowell and Sharpley 2001). Mean TC and TN also were lower in bed than bank sediments (Table 3). Mean TP and Mehlich-3-extractable P, however, were similar in bed and bank sediments. Total P concentrations in the bank sediments were similar to those found in stream bank soils throughout the Rathbun Lake watershed as reported by Tufekcioglu et al. (2012), but somewhat lower than the 360–555 mg P kg$^{-1}$ range that Zaimes et al. (2008) found in other watersheds in southeast Iowa. The range of TP concentrations in stream bed sediments collected by Zaimes et al. (2008) was also higher (389–964 mg P kg$^{-1}$) than we found in samples from the four sites (Table 3), but Zaimes et al. (2008) reported that samples had significant amounts of fine-textured material, which tend to have higher TP concentrations.

The amounts of Fe, Al, and Mn extracted from the bed and bank sediments varied among the sites, but the only statistical differences were between extractable Fe levels in the two types of sediments (Table 4). Higher levels of Mehlich-3-extractable Fe were measured in bank sediments than in bed sediments, whereas citrate-bicarbonate-dithionite (CBD)-extractable Fe levels were higher and oxalate-extractable Fe tended to be higher ($p = 0.053$) in bed sediments than in bank sediments. Although the Mehlich-3 and ammonium oxalate solutions both probably extract some of the poorly crystalline forms of Fe oxides from these sediments (Hongthanat et al. 2011), the results suggest that the oxalate and CBD extracts dissolve other forms of Fe as well. Many of the chemical properties of the bed and bank sediments were similar (Tables 2 and 3), which suggests that the bed sediments have not been transported from first-order streams within the watershed or adjacent upland soils. Source tracking (Tomer et al. 2010), however, was not utilized in this study.

Table 2  Water chemistry for four selected stream sites in the Rathbun Lake watershed

| Water chemistry | Sampling site | | | |
|---|---|---|---|---|
| | M[a] | H | B | P |
| DP[b] (mg L$^{-1}$) | 0.117 (0.015–0.315) | 0.145 (<0.010–0.292) | 0.106 (0.031–0.242) | 0.065 (<0.010–0.142) |
| Total P (mg L$^{-1}$) | 0.36 (0.10–0.76) | 0.40 (0.21–0.73) | 0.32 (0.13–0.98) | 0.30 (0.10–0.98) |
| TSS (mg L$^{-1}$) | 495 (180–1035) | 390 (175–730) | 470 (75–995) | 451 (285–965) |

Means are shown, with range in parentheses. Grab samples were collected biweekly from March until November of 2008 and 2009 ($n = 35$). Total suspended sediment (TSS) was measured March through August in 2009 ($n = 17$)

[a] Site M is Honey Creek; site H is West Jackson Creek; site B is Walker Creek; site P is Ninemile Creek

[b] Dissolved phosphorus

**Table 3** Physicochemical properties of stream bank and bed materials collected from four selected sites in the Rathbun Lake watershed

| Sediments[a] | pH | TC (g kg$^{-1}$) | TN | Sand | Silt | Clay | TP (mg kg$^{-1}$) | P$_{M3}$ | K$_{M3}$ | Ca$_{M3}$ | Mg$_{M3}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M-bed | 7.3 | 8.2 | 0.7 | 500 | 340 | 160 | 314 | 37 | 78 | 1946 | 364 |
| M-bank | 7.2 | 10.1 | 0.8 | 330 | 480 | 190 | 278 | 26 | 57 | 1959 | 353 |
| H-bed | 7.5 | 2.7 | 0.2 | 920 | 50 | 30 | 177 | 32 | 20 | 550 | 72 |
| H-bank | 6.5 | 10.1 | 0.9 | 350 | 490 | 160 | 284 | 36 | 77 | 1559 | 270 |
| B-bed | 8.0 | 3.2 | 0.1 | 820 | 120 | 60 | 454 | 28 | 37 | 1250 | 179 |
| B-bank | 6.5 | 11.9 | 0.9 | 290 | 540 | 180 | 209 | 25 | 62 | 1631 | 324 |
| P-bed | 8.2 | 2.3 | 0.1 | 940 | 40 | 20 | 314 | 17 | 12 | 654 | 68 |
| P-bank | 7.2 | 6.7 | 0.6 | 490 | 360 | 150 | 306 | 68 | 67 | 1817 | 309 |
| Bed mean | 7.8a[b] | 4.1b | 0.3b | 795a | 138b | 68b | 315a | 29a | 37a | 1100a | 171a |
| Bank mean | 6.9b | 9.7a | 0.8a | 365b | 468a | 170a | 269a | 39a | 66a | 1742a | 314a |

[a] Site M is Honey Creek; site H is West Jackson Creek; site B is Walker Creek; site P is Ninemile Creek

[b] Within each column, values with the same letter are not significantly different ($P \leq 0.05$)

## Indicators of phosphorus environmental risk and relation to water quality

By comparing several indicators of P loss risk, we can determine which indices can consistently identify high-risk sediments and thereby guide decisions about sediment and soil management along stream corridors and adjacent agricultural lands. The simplest index is the Mehlich-3 soil test. In Iowa, this test is an agronomic tool, so its use as an environmental threshold to predict the likelihood of P loss from soils or sediments has not yet been defined. In Arkansas, however, the environmental threshold for P$_{M3}$ has been set at 150 mg kg$^{-1}$ (Sharpley et al. 2003). According to this standard, the bank and bed sediments in the four streams leading to Rathbun Lake had a low risk of P loss (Table 3).

Degree of phosphorus saturation, estimated by either oxalate or Mehlich-3 extraction (Eqs. 1, 2, and 3), is a widely accepted indicator of the potential for environmental risk associated with soil or sediment P transport (Schoumans 2009). In principle, the higher the DPS value, the more easily P can be desorbed from sediment. The DPS estimated by ammonium

**Table 4** Elements released from four selected stream bank and bed sediments by Mehlich-3 (M3), ammonium oxalate (ox), or citrate-bicarbonate-dithionite (d) extraction

| Sediments[a] | Fe$_{M3}$ (mg kg$^{-1}$) | Al$_{M3}$ | P$_{ox}$ | Fe$_{ox}$ | Al$_{ox}$ | Mn$_{ox}$ | Fe$_d$ | Al$_d$ | Mn$_d$ |
|---|---|---|---|---|---|---|---|---|---|
| M-bed | 300 | 516 | 174 | 3135 | 755 | 670 | 6280 | 270 | 700 |
| M-bank | 358 | 311 | 163 | 2821 | 884 | 550 | 5300 | 240 | 560 |
| H-bed | 157 | 391 | 114 | 1788 | 250 | 110 | 2320 | 60 | 110 |
| H-bank | 446 | 686 | 170 | 2608 | 918 | 230 | 3890 | 220 | 260 |
| B-bed | 148 | 279 | 247 | 5596 | 425 | 2160 | 14,470 | 490 | 2200 |
| B-bank | 437 | 472 | 113 | 2327 | 1065 | 190 | 2600 | 180 | 210 |
| P-bed | 85 | 187 | 147 | 3164 | 234 | 1650 | 15,970 | 470 | 1730 |
| P-bank | 208 | 611 | 214 | 2112 | 492 | 250 | 3840 | 170 | 290 |
| Bed mean | 173b[b] | 343a | 170a | 3420a | 416a | 1148a | 9760a | 323a | 1185a |
| Bank mean | 362a | 520a | 165a | 2467a | 840a | 305a | 3907b | 203a | 330a |

[a] Site M is Honey Creek; site H is West Jackson Creek; site B is Walker Creek; site P is Ninemile Creek

[b] Within each column, values with the same letter are not significantly different ($P \leq 0.05$)

oxalate extraction (DPS-ox(Fe + Al)) was developed in the Netherlands for noncalcareous sandy soils, and a threshold value was set at 25 % (Schoumans 2009). If this value was defined as the threshold in the present study, none of the sediments had a high risk of P loss (Table 5). The DPS calculated from Mehlich-3-extractable Fe plus Al (DPS-M3(Fe + Al)) was developed for sandy loam and loamy sand soils in Delaware (Sims et al. 2002). For Delaware soils, the threshold value is 15 %. In Iowa, a threshold value of DPS-M3(Fe + Al) has not been defined, but if the Delaware threshold is used, the risk of P loss would be considered low for both the bed and bank sediments (DPS-M3(Fe + Al) <15 %). It should be noted that in these sediments, with near neutral pH and high Ca saturation, Al and Al oxides are unlikely to be significant participants in P retention or release (Hongthanat et al. 2011). The DPS calculated from Mehlich-3-extractable Ca (DPS-M3(Ca)) has been proposed for neutral to alkaline soils (Kleinman and Sharpley 2002, Ige et al. 2005), but a critical value has not been defined. Bed sediments from site H and bank sediments from site P had the highest values of DPS-M3(Ca), suggesting greater potential for P loss than other sediments, but similar to the other indices, values varied little among the sediments from the four sampling sites (Table 5). Regardless of calculation method, mean DPS values were similar for bed and bank sediments (Table 5), thereby providing little insight for P risk assessment.

Equilibrium P concentration, PEBC, and native P sorbed on solid phase ($S_0$) for the bed and bank sediments are also summarized in Table 5. The EPC values of the sediments ranged from 0.02 to 0.12 mg $L^{-1}$. This range is much smaller than that observed for DP concentrations in the streams (0.010 to 0.315 mg $L^{-1}$; Table 2); however, the EPC values were determined for sediments collected only under base-flow conditions. Under these conditions, the bed sediments would play a role in regulating DP concentration in the water column of these streams, as has been observed in several other studies (McDowell and Sharpley 2001; Haggard et al. 2007; McDaniel et al. 2009). The PEBC values of the sediments varied widely, ranging from 75 to 1754 L $kg^{-1}$. The difference of EPC, PEBC, and $S_0$ values between bed and bank sediments was not significant; however, mean PEBC for the bank sediments tended to be higher ($p = 0.19$) than that of the bed sediments, and bank sediments tended to have more ($p = 0.14$) native P ($S_0$) sorbed on the solid phase (Table 5).

At the four selected sites, the EPC values were used to determine whether bed sediments might behave as sources or sinks for P by comparing the values with DP in stream water. The status of bed sediments as a sink or a source of P to water is shown in Fig. 3 for 2009. Although bank sediments would only periodically be in contact with stream water, the status of these sediments as sinks or sources of P should be similar, given the EPC values of these materials (Table 5). Bed sediments at site H would behave as

Table 5　Degree of phosphorus saturation (DPS), calculated with ammonium oxalate and Mehlich-3 extraction data, equilibrium phosphorus concentration (EPC), phosphorus equilibrium buffering coefficient (PEBC), and native P sorbed on the solid phase ($S_0$) of stream bed and bank sediments from four selected sites in the Rathbun Lake watershed

| Sediments[a] | $DPS_{ox}(Fe + Al)$ (%) | $DPS_{M3}(Fe + Al)$ | $DPS_{M3}(Ca)$ | EPC (mg $L^{-1}$) | PEBC (L $kg^{-1}$) | $S_0$ (mg $kg^{-1}$) |
|---|---|---|---|---|---|---|
| M-bed | 7 | 5 | 2 | 0.05 | 361 | 18 |
| M-bank | 6 | 5 | 2 | 0.07 | 152 | 10 |
| H-bed | 9 | 6 | 8 | 0.08 | 89 | 7 |
| H-bank | 7 | 3 | 3 | 0.03 | 1271 | 34 |
| B-bed | 7 | 7 | 3 | 0.12 | 82 | 9 |
| B-bank | 5 | 3 | 2 | 0.02 | 1754 | 36 |
| P-bed | 7 | 7 | 3 | 0.12 | 75 | 9 |
| P-bank | 12 | 8 | 5 | 0.10 | 308 | 29 |
| Bed mean | 7a[b] | 6a | 4a | 0.09a | 153a | 11a |
| Bank mean | 7a | 5a | 3a | 0.06a | 871a | 28a |

[a] Site M is Honey Creek; site H is West Jackson Creek; site B is Walker Creek; site P is Ninemile Creek

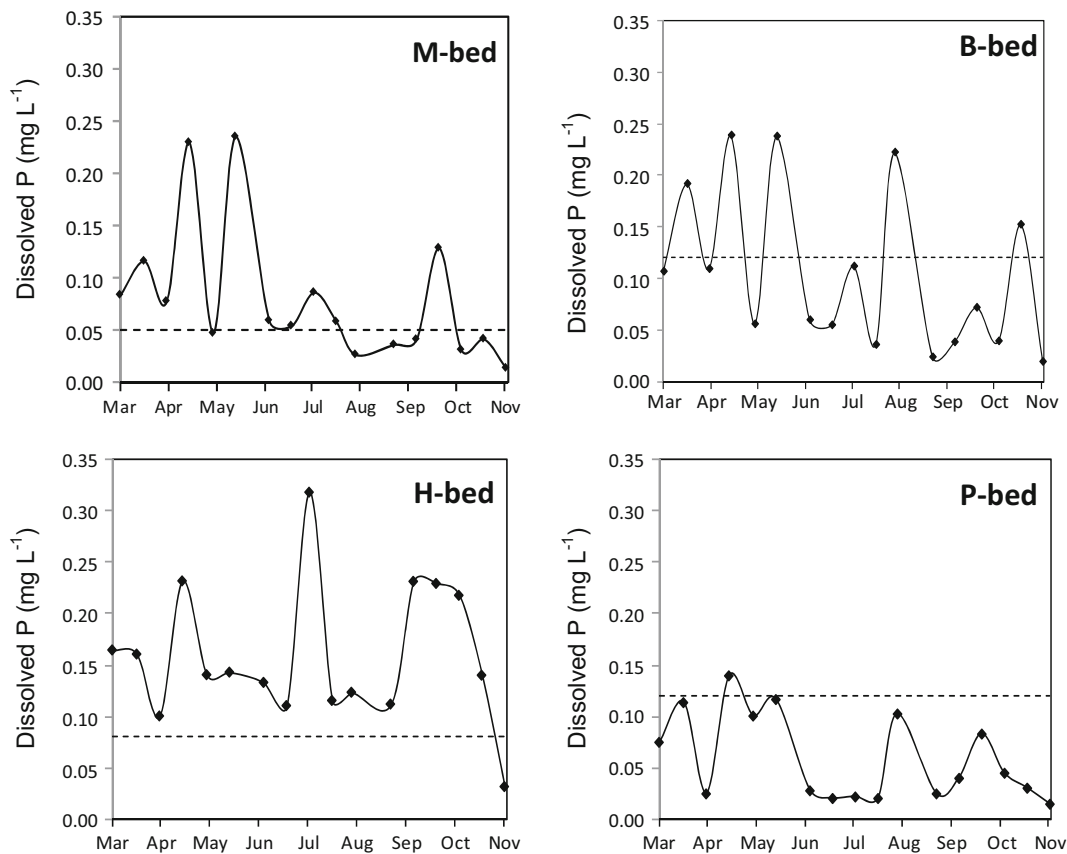[b] Within each column, values with the same letter are not significantly different ($P \leq 0.05$)

**Fig. 3** Dissolved P concentrations in stream water collected from each sampling site during March through November 2009. The *dashed lines* represent the equilibrium P concentration (EPC) of the individual bed sediments

a sink by retaining P from water in West Jackson Creek until November when the concentration of P in the stream water dropped below the EPC (Fig. 3). At this point, the sediment would release P into the water column to adjust to equilibrium. In contrast to H bed sediments, the EPC of bed sediments at site P was higher than that of site H, so bed sediments at site P would act as a source of P to water in Ninemile Creek (Fig. 3). Moreover, the concentration of P in the water column of Ninemile Creek was generally lower than that of West Jackson Creek (Fig. 3). For sites M and B (Honey Creek and Walker Creek, respectively), DP concentrations in the water column fluctuated throughout the year, so that the bed sediments were both sources and sinks for P several times during the March to November period. This suggests that the sediments in these streams act as a transient pool for P. Haggard et al. (1999) and McDaniel et al. (2009) found similar fluctuations in streams in Oklahoma and Illinois, respectively.

Relationship of sediment properties to equilibrium phosphorus concentration and phosphorus equilibrium buffering coefficient

We explored possible correlations between sediment properties and the EPC and PEBC values (Table 6). For this analysis, bank and bed sediment data were pooled. The strongest relationship was between EPC and $Fe_{M3}$ ($r^2 = 0.87$, $p < 0.001$). The correlation was negative ($r = -0.93$), indicating that as $Fe_{M3}$ levels increased, sediment EPC decreased, making P desorption less likely (Fig. 4). This result contrasts with that of McDowell and Sharpley (2001), as well as what was reported by Hongthanat et al. (2011), in which the EPC of the alluvial soil materials (as opposed to sediments) in the watershed was positively related to $Fe_{M3}$. Interestingly, the EPC of sediments was also negatively correlated with total C and total N (Table 6; Fig. 4), whereas $Fe_{M3}$ was positively correlated with total C ($r = 0.96$, $p < 0.001$) and total N ($r = 0.95$, $p < 0.001$) (data not shown). These results

**Table 6** Coefficients of determination ($r^2$), correlation coefficients ($r$), and levels of significance between equilibrium P concentration (EPC), P equilibrium buffering capacity (PEBC), and sediment physicochemical properties

| Property | EPC | | | PEBC | | |
|---|---|---|---|---|---|---|
| | $r^2$ | $r$ | $p$ value | $r^2$ | $r$ | $p$ value |
| pH | 0.85 | 0.92 | <0.01 | 0.71 | −0.84 | <0.01 |
| Total C | 0.74 | −0.86 | <0.01 | 0.56 | 0.75 | <0.05 |
| Total N | 0.75 | −0.87 | <0.01 | 0.50 | 0.71 | <0.05 |
| TP | 0.30 | 0.55 | 0.16 | 0.14 | 0.38 | 0.35 |
| $P_{M3}$ | 0.00 | 0.02 | 0.97 | 0.00 | 0.05 | 0.91 |
| $K_{M3}$ | 0.48 | 0.69 | 0.06 | 0.28 | 0.53 | 0.18 |
| $Ca_{M3}$ | 0.24 | 0.49 | 0.22 | 0.10 | 0.32 | 0.44 |
| $Mg_{M3}$ | 0.36 | 0.60 | 0.11 | 0.17 | 0.42 | 0.30 |
| $Na_{M3}$ | 0.40 | 0.63 | 0.09 | 0.20 | 0.45 | 0.27 |
| $Fe_{M3}$ | 0.87 | −0.93 | <0.001 | 0.65 | 0.81 | <0.05 |
| $Al_{M3}$ | 0.41 | 0.64 | 0.08 | 0.31 | 0.56 | 0.15 |
| Clay | 0.52 | −0.72 | <0.05 | 0.31 | 0.56 | 0.15 |
| Silt | 0.63 | −0.79 | <0.05 | 0.51 | 0.71 | <0.05 |
| Sand | 0.60 | 0.78 | <0.05 | 0.45 | 0.67 | 0.07 |
| $Fe_d$ | 0.55 | 0.74 | <0.05 | 0.23 | 0.48 | 0.22 |
| $Fe_{ox}$ | 0.55 | 0.74 | <0.05 | 0.20 | 0.44 | 0.27 |
| $Al_d$ | 0.34 | 0.58 | 0.13 | 0.10 | 0.32 | 0.44 |
| $Al_{ox}$ | 0.08 | 0.28 | 0.51 | 0.00 | 0.06 | 0.88 |
| $Mn_d$ | 0.51 | 0.72 | <0.05 | 0.22 | 0.47 | 0.23 |
| $Mn_{ox}$ | 0.51 | 0.71 | <0.05 | 0.23 | 0.48 | 0.23 |
| $P_{ox}$ | 0.57 | 0.76 | 0.03 | 0.20 | 0.45 | 0.26 |

suggest that Fe associated with organic matter, and readily extractable by the Mehlich-3 solution, could be responsible for P sorption by these sediments.

For the sediments from the four sites, there was a strong positive correlation between EPC and pH ($r = 0.92$, $p < 0.01$). This finding is similar to what was reported by Koski-Vähälä and Hartikainen (2001) who found that EPC increased with pH. Generally, an increase in soil pH causes the functional groups on the surface of clays and metal oxides to become more negatively charged (Pierzynski et al. 2005). As a result, P is more readily desorbed as pH increases.

Sediment particle size was also related to EPC (Table 6; Fig. 4). As sand content increased, EPC also increased ($r = 0.78$, $p < 0.05$). This is reasonable as there would be less clay-sized particles to sorb P. While sand-sized quartz did not strongly retain P, fine sediments did, as indicated by the negative relationship between EPC

and clay content ($r = −0.72$, $p < 0.05$). Sediment EPC was also negatively related to silt content ($r = −0.79$, $p < 0.05$), which contrasts with the results of Haggard et al. (1999) who found that EPC increased with silt content ($r = 0.91$, $p < 0.01$). We can speculate that the silt-sized minerals and their ability to sorb P differed between southern Iowa and the sites in Oklahoma where Haggard et al. (1999) conducted research. In Illinois streams, McDaniel et al. (2009) found that EPC values were negatively correlated ($r = −0.49$, $p < 0.0001$) with the silt plus clay fraction of sediments, which tends to support our results.

There also were positive relationships between EPC and $Fe_d$, $Fe_{ox}$, $Mn_d$, $Mn_{ox}$, and $P_{ox}$ (Table 6). However, these significant relationships were based solely on data from the bed sediments of sites B and P. Bed sediments from these two sites had the highest EPC values, as well as the highest concentration of $Fe_d$, $Fe_{ox}$, $Mn_d$, $Mn_{ox}$, and $P_{ox}$. If data from these two sites are excluded, the relationships no longer exist.

Phosphorus equilibrium buffering capacity was negatively correlated to pH ($r = −0.84$) and positively related to total C ($r = 0.75$), total N ($r = 0.71$), $Fe_{M3}$ ($r = 0.81$), and silt content ($r = 0.71$) (Table 6). However, these relationships were essentially driven by the same bed sediment data from sites B and P. Therefore, PEBC is not well described by any of the measured sediment properties.

## Summary and conclusions

This research, part of a larger study of the effects of livestock grazing on stream bank erosion and P losses in the Rathbun Lake watershed, focused on the interaction of P in stream sediments and P in the water column at four subcatchment sites. Stream water samples collected from locations throughout the watershed indicated that median DP concentrations ($0.07$ mg $L^{-1}$) were comparable to those reported in other central and southern Iowa studies, while median TP ($0.24$ mg $L^{-1}$) concentrations were high relative to the proposed nutrient criteria value ($0.10$ mg TP $L^{-1}$) for wadeable, warm-water streams in Iowa. At the four sites where sediment samples were collected, stream water DP concentrations ranged from $0.065$ to $0.145$ mg P $L^{-1}$, while TP concentrations ranged from $0.30$ to $0.40$ mg P $L^{-1}$. These high stream water P concentrations suggest that some form of remediation is necessary to improve water quality in Rathbun Lake.

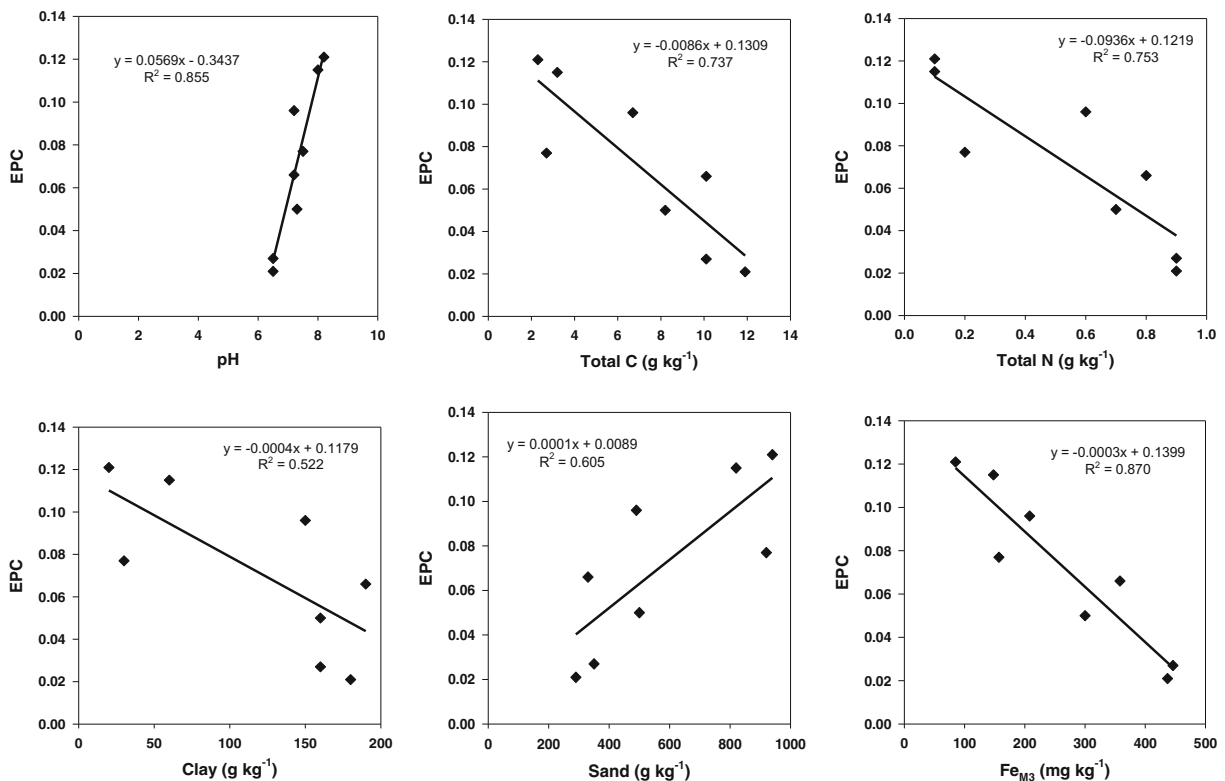Phosphorus mobility in streams is regulated by a number of interacting biotic and abiotic processes that

**Fig. 4** Relationships between equilibrium P concentration (EPC) and pH, total C, total N, clay, sand, and Mehlich-3-extractable Fe (Fe$_{M3}$) for bank and bed sediments collected from four locations in the Rathbun Lake watershed

make both prediction and control challenging. In some cases, sediments alone are thought to exert significant control of water-column P, while in others, sediments in combination with microbes and macrophytes act to buffer stream P loads (Haggard et al. 2007; McDaniel et al. 2009). Sediment P content, readiness to release P to the stream (that is, to act as a source of P), and ability to retain soluble P (that is, act as a sink for P) may vary significantly. To assess the risk of P release from sediments in the Rathbun Lake watershed, we focused on three P sorption–desorption indices. Results suggested that the stream bank and bed sediments had low risk of P release when evaluated by Mehlich-3-extractable P and the DPS. However, the EPC indicated that some bed sediments could release P to water depending on DP concentrations in the stream water column, which varied among the sites and with the time of year. The likelihood of P desorption from the sediments increased with increasing pH and sand content, but decreased with Fe associated with organic matter. There is still uncertainty about the role of Fe in P sorption–desorption for the soils and sediments in this watershed. Because the EPC values of the bed sediments at the four selected sampling sites exceeded DP concentrations in the corresponding streams at some point during the year, changes in land use within the riparian areas may, at least initially, have little effect on P concentrations in these and other streams leading to Rathbun Lake. As recently pointed out by Jarvie et al. (2013) and Sharpley et al. (2013), the release of "legacy" P from stream sediments can buffer against rapid water quality improvement after nutrient management measures are introduced. On the other hand, the low concentrations of DP relative to TP in these streams suggest that if P inputs from eroded bank sediments are reduced, whether by grazing management, stream bank stabilization, or some other practice, P loads to Rathbun Lake can be reduced as well.

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