

## Variations in Stream Water and Sediment Phosphorus among Select Ozark Catchments

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Stream sediments play a large role in the transport and fate of soluble reactive phosphorus (SRP) in stream ecosystems, and equilibrium P concentrations ( $EPC_0$ ) of benthic sediments at which P is neither adsorbed nor desorbed are often related to stream water SRP concentrations. This study evaluated (i) the variation among water chemistry and sediment-P interactions among streams draining catchments that varied in the land use; (ii) the relations between SRP concentration, sediment  $EPC_0$ , and other measured abiotic factors (e.g., particle size distribution, slope of linear sorption isotherms, etc.) in the stream sediments; and (iii) the use of the traditional Mehlich-3 (M3) soil extraction on stream sediments to elucidate other abiotic factors (e.g., M3P, P saturation ratio, etc.) related to SRP concentration in stream sediments. Stream water and sediments were sampled at 22 selected Ozark streams in northwest Arkansas during fall 2003 and spring 2004. Nitrate-N concentrations in the water column ( $r = 0.69$ ) and modified P saturation ratios ( $PSR_{mod}$ ) of the benthic sediments ( $r = 0.79$ ) at the selected streams increased with an increase in percent pasture in the catchments, whereas SRP concentration ( $r = -0.56$ ) and Mehlich-3-extractable P (M3P) content ( $r = -0.47$ ) decreased with an increase in the percent forested area. Soluble reactive P concentrations in the stream water were positively correlated to sediment  $EPC_0$  ( $r = 0.51$ ), although sediment  $EPC_0$  was generally greater than SRP. The M3 soil extraction was useful in identifying abiotic factors related to SRP concentrations in the selected streams, in particular SRP concentrations were positively correlated to M3P contents ( $r = 0.50$ ) and  $PSR_{mod}$  ( $r = 0.71$ ) of the benthic sediments. Thus, M3P and  $EPC_0$  estimates from stream sediments may be valuable yet simple indicators of whether benthic sediments act as sinks or sources of P in fluvial systems, as well as estimating changes in stream SRP concentrations.

SEVERAL studies have evaluated factors controlling soluble reactive P (SRP) concentrations in streams, particularly the ability of stream sediments to provide a dissolved P buffering mechanism. Stream sediments have the ability to adsorb dissolved P from the water column or release P to overlying waters depending on the equilibrium exchange concentration, or sediment equilibrium P concentration ( $EPC_0$ ; Froelich, 1988). The sediment  $EPC_0$  is the concentration in the aqueous phase (e.g., water column in streams) at which net adsorption or desorption of dissolved P from the benthic sediment does not occur (Taylor and Kunishi, 1971; Klotz, 1988), i.e., the aqueous and solid phases are in a dynamic equilibrium with respect to dissolved P. The sediment  $EPC_0$  concept is often used to determine if benthic sediments are a potential source or sink of dissolved P to the overlying waters in streams.

A myriad of factors influence the dissolved P equilibrium concentration between the water column and benthic sediments in streams, including biotic processes and various abiotic characteristics. Important abiotic characteristics revolve around the ability of stream sediments to adsorb dissolved P from the water column, such as benthic sediment particle size distribution (Klotz, 1988; Haggard et al., 1999), the content of water-extractable or other P forms in the sediments (McDowell et al., 2002; Ekka et al., 2006), divalent cations in aqueous solution (Klotz, 1991) and exchangeable in sediments, and the strength with which dissolved P is adsorbed to sediments, among other potential factors. The influence of sediments on stream water P concentrations is similar to that observed in soil-water systems, where dissolved P concentrations in runoff water from small plots are positively correlated to water-extractable P and Mehlich-3 P (M3P) content of soils (Pote et al., 1999; Vadas et al., 2005).

Dissolved P equilibrium concentrations between stream sediments and water can also be affected by external factors, such as municipal wastewater treatment plant (WWTP) effluent discharges high in dissolved P (Popova et al., 2006; Ekka et al., 2006). Biotic

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**Abbreviations:** ANOVA, analysis of variance; Chl-a, chlorophyll-a; DO, dissolved oxygen;  $EPC_0$ , equilibrium P concentration; LSD, least significant difference; M3, Mehlich-3 extraction; M3P, Mehlich-3 P; Max, maximum; Med, median; Min, minimum;  $NH_4-N$ , ammonium-N;  $NO_3-N$ , nitrate N;  $NO_2-N$ , nitrite N; PSR, P saturation ratio; Q, stream discharge; SRP, soluble reactive P; SD, standard deviation; TN, total N; TP, total P;  $u$ , water velocity; WWTP, wastewater treatment plant.

Table 1. Catchment area, percent of forested, pasture and urban land use and land use classification of the selected Ozark streams, Northwest Arkansas, USA.

Selected stream sites†	Catchment area ha	Land use classification‡			
		Forest %	Pasture %	Urban %	
Hock Creek (8)	3,835	70.5	29.3	0.2	forest
Lee Creek(6)	4,964	90.0	10.0	<0.1	forest
Lollars Creek(19)	11,217	79.3	20.6	<0.1	forest
Ballard Creek (3)	10,476	49.0	49.9	0.9	mixed
Baron Fork (5)	9,076	52.6	46.8	0.6	mixed
Brush Creek (7)	4,682	53.9	46.1	<0.1	mixed
Clear Creek (1)	2,962	40.7	59.2	<0.1	mixed
Coon Creek (12)	3,563	47.1	52.9	<0.1	mixed
Flint Creek (13)	4,822	44.2	55.7	0.1	mixed
South Spavinaw Creek (9)	4,038	58.9	41.0	<0.1	mixed
Beaty Creek (11)	2,185	14.5	85.5	<0.1	pasture
Cincinatti Creek (15)	10,264	35.1	64.9	<0.1	pasture
Glade Creek (2)	5,216	35.2	64.7	0.1	pasture
Little Osage Creek (14)	9,308	13.9	81.0	4.9	pasture
Spavinaw Creek (10)	6,417	39.1	60.8	0.1	pasture
Wildcat Creek (17)	3,166	34.2	65.8	<0.1	pasture
Goose Creek (16)	3,213	36.4	53.7	9.8	urban
Hamestring Creek (18)	2,651	28.8	51.4	19.7	urban
Moores Creek (4)	934	18.2	68.0	13.6	urban
Mud Creek Tributary (21)	293	57.7	<0.1	42.3	urban
Skull Creek (22)	169	65.9	<0.1	33.8	urban
Town Branch (20)	122	38.7	<0.1	60.9	urban

† Site no. in Fig. 1.

‡ Land use classification definitions are as follows: *Forest* catchments were those that had more than 60% forested land use and < 10% urban land use; *Pasture* catchments were those that had more than 60% pasture land use and < 10% urban; and *Urban* catchments were those that had approximately 10% or more urban development. The *Mixed* catchments did not fit into any of the other land use categories, and these catchments generally had a close to equal mix between forested and pasture land use.

processes in benthic sediments are extremely important in the uptake and release of dissolved P (Gächter and Meyer, 1993) and often account for a large fraction (more than 38%) of dissolved P adsorbed from aqueous solutions (e.g. see Haggard et al., 1999; Khoshmanesh et al., 1999). The importance of biotic processes is further supported by interactions among sediment EPC<sub>0</sub>, benthic organic matter content (Smith et al., 2005), and alkaline phosphatase activity in the benthos (Klotz, 1985).

It is clear that uptake and release of P by stream sediments are important components in the fate and transport of dissolved P through fluvial systems and in the modification of P export from catchments (House and Warwick, 1998; McDowell et al., 2003). Many studies have evaluated sediment and dissolved P interactions in individual streams (e.g., Klotz, 1988, 1991; House and Warwick, 1998; McDowell et al., 2002) or on a small number of streams in a particular region and similar land use types (e.g., Haggard et al., 1999; Ekka et al., 2006). However, few studies have evaluated sediment EPC<sub>0</sub> and potential abiotic regulating factors over a large number of streams (greater than 20) draining catchments with differing proportions of forest, pasture, and urban development. Thus, the main objective of our study was to evaluate the interactions of dissolved P between stream water and sediments at 22 streams in the Ozark Highlands of northwest

Arkansas, USA. Specifically, we focused on variations in water chemistry and sediment P parameters with catchment land uses (Objective 1), the relation between water column dissolved P concentrations and sediment EPC<sub>0</sub> at the selected streams (Objective 2), and the use of the traditional Mehlich-3 (M3; Mehlich, 1984) soil extraction to elucidate factors related to dissolved P concentrations in streams and benthic sediment EPC<sub>0</sub> (Objective 3).

## Materials and Methods

### Study Site Description

We selected one site on 22 different streams in the Ozark Highlands of Benton, Washington, and Madison Counties in Northwest Arkansas, USA (Table 1, Fig. 1). These streams drained catchments of various sizes (122 to 11200 ha) and multiple land uses, including forest, pasture, and urban development. ArcGIS software (ESRI GIS and Mapping Software, St. Charles, MO) was used to determine catchment area and land use classifications for each of the water quality and sediment sampling sites; land use was determined using 1999 land use and land cover data layers from the University of Arkansas Center for Advanced Spatial Technology (CAST, 2006). The land use and land cover information was classified into four categories: forest, pasture, urban, and transitional areas or barren lands. The selected sites drained catchments that varied from ~90% forest to ~86% pasture to ~61% urban, and barren and transitional areas were always the smallest fraction (less than 5%) of designated land use in each of the catchments. The 22 selected streams were separated into land use categories (Table 1), which were designated as: *Forest*, *Mixed*, *Pasture*, and *Urban*. The *Forest* catchments were those that had more than 60% forested land use and less than 10% urban land use; *Pasture* catchments were those that had more than 60% pasture land use and less than 10% urban; and *Urban* catchments were those that had approximately 10% or more urban development. The *Mixed* catchments did not fit into any of the other land use categories, and these catchments generally had a close to equal mix between forested and pasture land use. We acknowledge that the separation of the selected catchments into these designated land use categories is somewhat arbitrary, but this was done to facilitate comparisons between selected streams. The 22 selected sites were sampled in late summer and fall 2003 (late August through early November) and in spring 2004 (mid May through mid June), and sampling dates were selected when the stream was in seasonal base flow conditions. Seasonal base flow discharge shows natural temporal and spatial variation in the Ozarks, and base flow conditions were preferred because benthic sediment control or maintenance of stream water SRP concentrations would occur during these low flow periods (McDowell et al., 2002).

### Field Methods

Water and sediment samples were collected at three points along the stream continuum, where water samples were collected in the thalweg of the defined stream channel, and sediment samples were collected from the top 5 to 10 cm along a transect per-

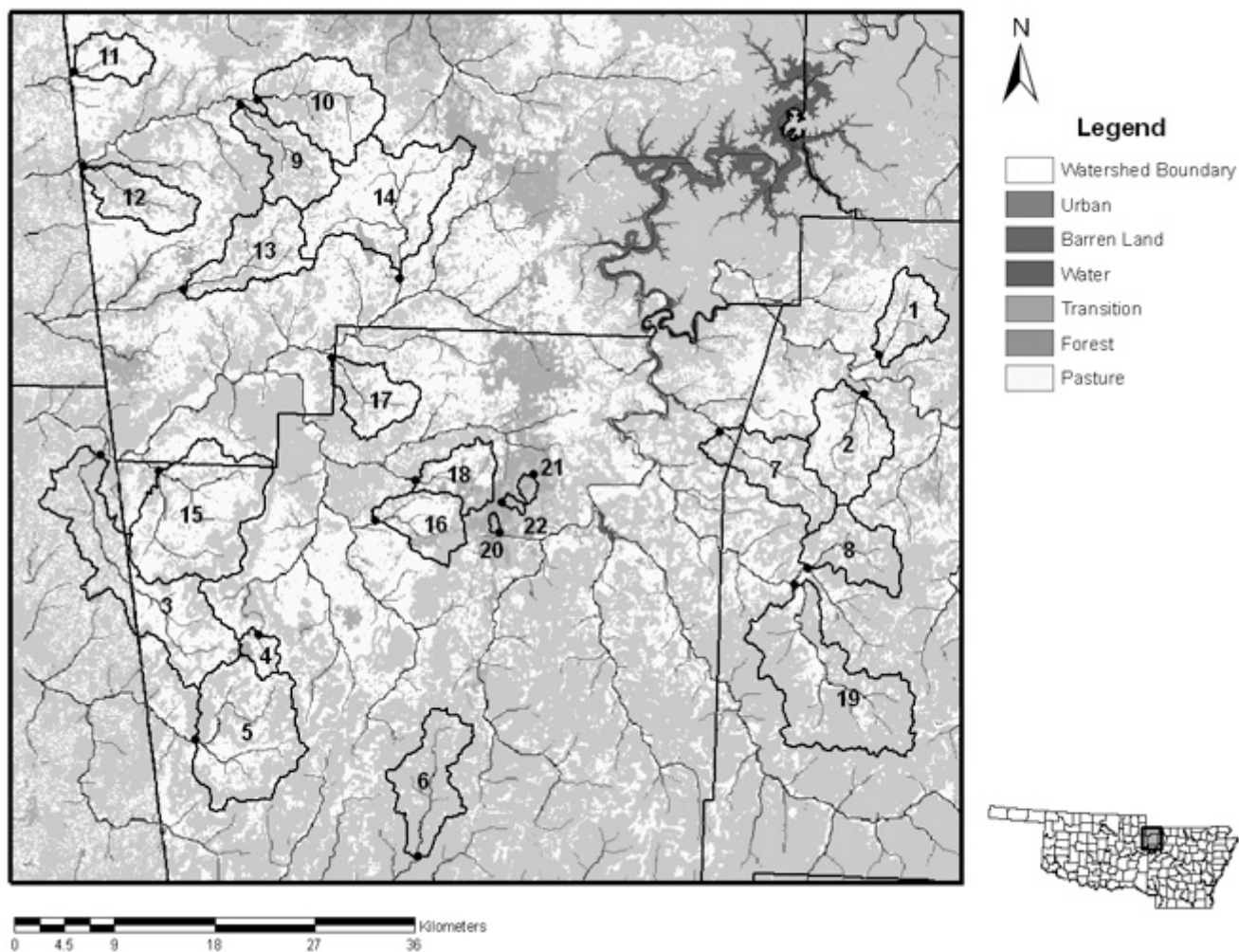


Fig. 1. Map delineating catchment area and land use distribution of the selected study sites on 22 different streams in the Ozark Highlands of Northwest Arkansas, USA. Note: Site numbers correspond to the numbers listed in parentheses in Table 1.

pendicular to stream flow until approximately 2 L in volume was collected. The gravel and cobble stream bottoms in typical Ozark streams have relatively large transient storage areas (see Haggard et al., 2001), where surface water interacts with bottom sediments often much deeper than 5 to 10 cm. Three individual water samples and three different composite sediment samples were collected at the selected sites along a stream reach that varied from 50 to over 100 m in distance. Water samples collected for chemical analyses included an unfiltered sample (~250 mL), a filtered acidified sample (~20 mL, 0.45  $\mu\text{m}$  membrane, syringe filtration,  $\text{pH} < 2$ ), and a filtered unacidified sample (~20 mL, 0.45  $\mu\text{m}$  membrane, syringe filtration); two other unfiltered water samples (~1 L each) were collected at each point for sestonic chlorophyll-a (Chl-a) analysis and also the sediment extractions. We measured pH (pH Testr 3, Oakton Instruments, West Cladwell, NJ), dissolved oxygen (DO) (YSI Model 85, Yellow Springs, OH), and conductivity and temperature (Orion Meter 115A plus, Beverly, MA) at the middle site. Water velocity ( $u$ ) was measured at the middle site along a transect divided into equally spaced intervals (Flo-Mate 2000, Marsh-McBirney, Inc., Frederick, MD), and depth was measured at the mid-point of each interval. Discharge ( $Q$ ) was estimated as

the sum of  $u$  multiplied by the cross-sectional area ( $A^2$ ) of each interval along each transect.

### Laboratory Methods

After sample processing in the field, all water samples for chemical analyses were frozen until specific analyses were completed. Filtered, acidified samples were analyzed for SRP using the automated ascorbic acid method (APHA, 1998) and ammonium-nitrogen ( $\text{NH}_4\text{-N}$ ) using the sodium nitropruside and salicylate method (APHA, 1998) on a Skalar San Plus Wet Chemistry Autoanalyzer (Skalar, the Netherlands). Nitrite N ( $\text{NO}_2\text{-N}$ ), nitrate N ( $\text{NO}_3\text{-N}$ ), and  $\text{Cl}^-$  were measured on filtered, unacidified samples using colorimetric determination of  $\text{NO}_2\text{-N}$  (APHA, 1998), Cd-Cu reduction of  $\text{NO}_3\text{-N}$  and subsequent colorimetric analysis of  $\text{NO}_2\text{-N}$  (APHA, 1998), and the mercuric thiocyanate reaction for  $\text{Cl}^-$  (Skalar Method, the Netherlands) on the autoanalyzer. A known volume of water (1 L) was filtered through a Whatman GF/F glassfiber filter, and the glassfiber filter was frozen until Chl-a analysis could be performed. The frozen filter was placed in a glass vial with 5 mL of aqueous acetone saturated with  $\text{MgCO}_3$  and then shredded.

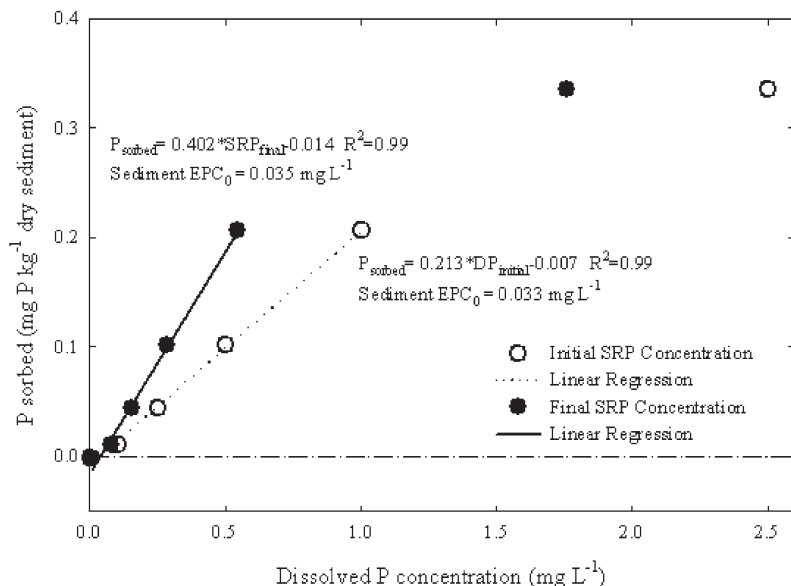


Fig. 2. Graphical display of the amount of phosphorus adsorbed by the sediment (P sorbed) as a function of initial and final soluble reactive phosphorus (SRP) concentration in the aqueous solution. Note: The linear regression of P sorbed and final SRP concentration was used to estimate the slope of the relation ( $K_{\text{slope}}$ ) and sediment equilibrium P concentration ( $EPC_0$ ) in this study, and the data point from the highest level of P enrichment, i.e., ambient SRP concentrations plus an additional  $2.5 \text{ mg PO}_4\text{-P L}^{-1}$ , was not used in the linear regression used to determine  $K_{\text{slope}}$  and sediment  $EPC_0$  in this example.

The glass vial was centrifuged and then the supernatant analyzed for Chl-*a* using the trichromatic method (APHA, 1998).

After return to the laboratory, sediments were immediately sieved through a 4.5-mm sieve, and particles less than 4.5 mm were used in the following extraction procedure. Stream water ( $\sim 1 \text{ L}$ ) was filtered through a 0.45- $\mu\text{m}$  membrane and used in the subsequent extractions as defined. Approximately 25 g of fresh, wet sediment were placed into a 125 mL Erlenmeyer flask, and then 100 mL of different solutions were added to each flask. Our solutions were comprised of filtered (0.45  $\mu\text{m}$ ) stream water spiked with additional amounts of  $\text{PO}_4\text{-P}$  at rates of 0.00, 0.10, 0.25, 0.50, 1.00, and  $2.50 \text{ mg L}^{-1}$ . For example, if the stream water had an SRP concentration of  $0.01 \text{ mg L}^{-1}$ , then our series of solutions would have initial SRP concentrations of 0.01, 0.11, 0.26, 0.51, 1.01, and  $2.51 \text{ mg L}^{-1}$ . The sediment slurry was shaken in a reciprocating type shaker for 1 h, and then allowed to settle for approximately 30 min. A 15- to 20-mL aliquot was filtered through a 0.45- $\mu\text{m}$  membrane, and then analyzed for SRP as previously described. The remaining sediment slurry was transferred into Al pans and dried for  $\sim 48 \text{ h}$  at  $80^\circ\text{C}$  to determine sediment dry mass. Simple linear regression of P sorbed ( $\text{mg P}_{\text{sorbed}} \text{ kg}^{-1}$  dry sediment) against final SRP concentration ( $\text{SRP}_{\text{final}}$ ,  $\text{mg L}^{-1}$ ) in the solution was used to estimate sediment  $EPC_0$ , where the  $x$  intercept represents the point of negligible P adsorption or release from sediments to the aqueous solution (Fig. 2). The slope ( $K_{\text{slope}}$ ) of this line was used as a measure of the ability of stream sediments to adsorb P, where greater  $K_{\text{slope}}$  values would indicate a stronger ability to adsorb P from the aqueous solution (Froelich, 1988).

In this study, we used fresh, wet sediments in our sorption studies because previous studies have shown that sediment drying

affects P sorption characteristics of sediment (e.g., see Baldwin, 1996; Watts, 2000) and the estimation of sediment  $EPC_0$ . We also used filtered (0.45  $\mu\text{m}$ ) stream water as the basis for our series of P solutions, because several studies (e.g., Klotz, 1988; House and Denison, 2000) have shown that dissolved cations (i.e., Ca, Mg, Fe, and Mn) can influence sediment  $EPC_0$  measurement. In fact, Popova et al. (2006) showed that sediment  $EPC_0$  was significantly less when determined using a  $\text{CaCl}_2$  solution of similar conductivity to the natural stream water than when using filtered stream water, as in this study.

In spring 2004, sediment samples from the 22 selected streams were also extracted using the M3 (Mehlich, 1984) method of soil analysis. The M3 extracts were analyzed using inductively coupled plasma-optical emission spectrometry (ICP-OES) to determine M3-extractable Al, As, B, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, S, Se, Ti, and Zn. The M3 elemental contents are reported on a  $\text{mg element kg}^{-1}$  dry sediment basis. We used the M3 data to determine the P saturation ratio (PSR) of these stream sediments (see Sims et al., 2002):

$$PSR = M3P / [M3Al + M3Fe] \quad [1]$$

However, we also calculated a modified PSR ( $PSR_{\text{mod}}$ ) based on the results obtained in this study that showed M3Fe, M3Mn, and M3Mg were important factors related to M3P in the sediment extracts (see Results and Discussion):

$$PSR_{\text{mod}} = M3P / [M3Fe + M3Mn + M3Mg] \quad [2]$$

Phosphorus saturation ratios were calculated on a molar basis using  $\text{mmol kg}^{-1}$  dry sediment.

## Data Analyses and Comparisons

The intent of our study was to focus on broad comparisons and correlations across the 22 selected Ozark streams. Therefore, we used the geometric mean of the data when collected in replicates, where the replicates represent different water or sediment samples collected at the separate transects. The geometric mean was used to minimize the influence of extreme values, and it is common to use log transformations in water quality data analyses and comparisons (Hirsch et al., 1991). We used a paired  $t$ -test of the data from fall 2003 and spring 2004 to ascertain any seasonal differences in parameters across the 22 selected streams, and we used simple linear or stepwise linear regression analyses to evaluate relations between two or more parameters. The strength of associations discussed in the text (see Results and Discussion) are quantified by correlation coefficients ( $r$ ) due to uncertain causality, whereas coefficients of determination ( $R^2$ ) are reported in figures as part of the presentation of the regression models. We used analysis of variance (ANOVA) with mean separation (least significant difference, LSD) to determine if any parameter varied among the four defined land use categories. All statistical compari-

sons were made using an a priori significance level of 0.05 and Statistix 8.0 software (Analytical Software, Tallahassee, FL).

## Results and Discussion

### Stream Flow and Water Chemistry

Stream  $Q$  was highly variable among the 22 selected sites on these Ozark streams (Table 2 and 3), ranging from 3 L s<sup>-1</sup> at Moores Creek to ~890 L s<sup>-1</sup> at Little Osage Creek in fall 2003 and from 2 L s<sup>-1</sup> at Mud Creek Tributary to ~1900 L s<sup>-1</sup> at Little Osage Creek in spring 2004. Stream  $Q$  increased significantly with an increase in catchment area ( $r = 0.63$ ,  $P < 0.01$ ), and  $Q$  was also positively correlated with  $u$  ( $r = 0.64$ ,  $P < 0.01$ ), which varied from a combined fall 2003 and spring 2004 average of 0.04 m s<sup>-1</sup> at Moore's Creek to 0.68 m s<sup>-1</sup> at Flint Creek. Stream  $Q$  and  $u$  were significantly greater (Paired  $t$ -test,  $P < 0.001$ ) in spring 2004 compared to fall 2003 across the selected streams, likely because seasonal base flow discharge was greater in the spring season due to increased precipitation and replenishing of alluvial ground waters over the winter and spring seasons which contribute to seasonal base flow.

Water chemistry and temperature varied between the seasons sampled where pH, conductivity, and DO were greater during base flow conditions in fall 2003, but water temperature and NH<sub>4</sub>-N, NO<sub>2</sub>-N, and NO<sub>3</sub>-N concentrations were greater in spring 2004 (Table 2 and 3). However, conductivity and sestonic Chl-a, SRP, TP, and Cl concentrations did not vary between the seasons across the selected sampling sites. Overall, the supply of N (i.e., NH<sub>4</sub>-N and NO<sub>3</sub>-N) at the selected streams was much greater than that of P (i.e., SRP), because molar N/P supply ratios ranged from 10 to 368. Studies in the southwestern Ozark Highlands have shown that periphytic algal growth in Ozark streams is often limited by the supply of P relative to N or is co-limited by the supply of N and P, when nutrients are the primary limiting factor for periphytic algal growth (Matlock et al., 1999; Popova et al., 2006). However, sestonic Chl-a concentrations were not correlated to the supply of either N and or P at the selected Ozark streams and were generally very low (<1 µg L<sup>-1</sup>), except at the slowly flowing Moore's Creek (1.2–21.2 µg L<sup>-1</sup>). In contrast, Lohman and Jones (1999) showed that sestonic Chl-a concentrations were positively correlated to the nutrient supply in northern Ozark streams, although these northern Ozark streams were generally larger than those sampled in our study (i.e., catchment area was much greater).

Average NO<sub>3</sub>-N concentrations increased with an increase in stream  $Q$  across the selected streams (data not shown,  $r = 0.57$ ,  $P < 0.01$ ), but it appears that catchment land use has the greatest influence on stream nutrient concentration and conductivity. Average NO<sub>3</sub>-N concentrations increased with an increase in the percent pasture land use within the catchment ( $r = 0.69$ ,  $P < 0.001$ ) (Fig. 3B), and NO<sub>3</sub>-N concentrations were greatest (ANOVA LSD,  $P < 0.001$ ) in the streams draining catchments in the *Pasture* and *Mixed* land use categories (Fig. 4B). On the other hand, average SRP and TP concentrations decreased with an increase in the percent forested land use within the catchment (SRP:  $r = -0.56$ ,  $P < 0.01$ ; TP:  $r = -0.53$ ,  $P < 0.05$ ) (Fig. 3A), and SRP concentrations were least in the streams draining catchments in the *Forest*

Table 2. Data summary of water chemistry, sediment-phosphorus sorption, and benthic particle size distribution (limited to particles < 4.5 mm in diameter) across the 22 selected sampling sites at Ozark streams in fall 2003 (late August through early November).

Parameter (units)	<i>n</i>	Min†	Med	Max	Mean	SD
pH	22	7.6	8.0	9.5	8.1*	0.4
Temperature (°C)	22	8.8	16.7	23.0	16.8*	3.9
Conductivity (µS cm <sup>-1</sup> )	22	91	332	472	313	106
DO‡ (mg L <sup>-1</sup> )	22	5.5	8.7	10.7	8.4*	1.5
Chlorophyll-a (µg L <sup>-1</sup> )	22	0.09	0.45	1.45	0.62	0.40
$Q$ (discharge, L s <sup>-1</sup> )	22	3	67	887	148*	206
$u$ (velocity, m s <sup>-1</sup> )	22	0.01	0.15	0.52	0.18*	0.15
SRP (mg L <sup>-1</sup> )	22	0.006	0.020	0.055	0.024	0.013
TP (mg L <sup>-1</sup> )	22	<0.010	0.030	0.091	0.036	0.024
NH <sub>4</sub> -N (mg L <sup>-1</sup> )	22	<0.01	0.04	0.10	0.04*	0.02
NO <sub>2</sub> -N (mg L <sup>-1</sup> )	22	<0.001	0.002	0.010	0.003*	0.002
NO <sub>3</sub> -N (mg L <sup>-1</sup> )	22	0.04	1.95	2.93	1.52*	1.11
Cl (mg L <sup>-1</sup> )	22	3.2	8.7	17.5	9.3	3.7
Sediment EPC <sub>0</sub> (mg L <sup>-1</sup> )	22	<0.001	0.049	0.298	0.095	0.095
K <sub>slope</sub> (L kg <sup>-1</sup> )	22	0.07	0.42	2.20	0.61*	0.53
Pebbles (%; 2.0–4.5 mm)	22	33.5	55.9	83.8	55.9	10.5
Sand (%; 0.05–2.0 mm)	22	12.5	36.5	57.2	37.0	9.2
Silt (%; 0.002–0.05 mm)	22	0.2	3.8	14.6	4.6	3.4
Clay (%; <0.002 mm)	22	<0.1	0.8	4.7	1.0	1.0

† Min denotes minimum value; Med denotes median value; Max denotes maximum value; Mean denotes the arithmetic average; SD denotes standard deviation; and \* denotes significant difference between fall 2003 and spring 2004 data using a paired  $t$ -test ( $P < 0.05$ ).

‡ DO, dissolved oxygen; SRP, soluble reactive P; TP, total P; EPC<sub>0</sub>, equilibrium P concentration.

Table 3. Data summary of water chemistry, sediment-phosphorus sorption, and benthic particle size distribution (limited to particles < 4.5 mm in diameter) across the 22 selected sampling sites at Ozark streams in spring 2004 (May through June).

Parameter (units)	<i>n</i>	Min†	Med	Max	Mean	SD
pH	22	6.7	7.5	8.0	7.5*	0.3
Temperature (°C)	22	14.2	20.0	24.3	19.4*	2.6
Conductivity (µS cm <sup>-1</sup> )	22	106	299	779	317	138
DO‡ (mg L <sup>-1</sup> )	22	4.9	7.8	10.2	7.8*	1.4
Chlorophyll-a (µg L <sup>-1</sup> )	22	0.18	0.62	21.16	1.62	4.38
$Q$ (discharge, L s <sup>-1</sup> )	22	2	284	1913	486*	497
$u$ (velocity, m s <sup>-1</sup> )	22	0.01	0.37	0.99	0.40*	0.27
SRP (mg L <sup>-1</sup> )	22	0.003	0.034	0.072	0.034	0.020
TP (mg L <sup>-1</sup> )	22	<0.010	0.036	0.215	0.043	0.044
NH <sub>4</sub> -N (mg L <sup>-1</sup> )	22	<0.01	0.02	0.07	0.02*	0.02
NO <sub>2</sub> -N (mg L <sup>-1</sup> )	22	0.003	0.006	0.024	0.007*	0.005
NO <sub>3</sub> -N (mg L <sup>-1</sup> )	22	0.05	2.75	5.34	2.54	1.66
Cl (mg L <sup>-1</sup> )	22	2.7	7.7	25.0	9.0	4.8
Sediment EPC <sub>0</sub> (mg L <sup>-1</sup> )	22	0.009	0.131	0.329	0.140	0.095
K <sub>slope</sub> (L kg <sup>-1</sup> )	22	0.15	0.28	0.86	0.34*	0.18
Pebbles (%; 2.0–4.5 mm)	22	29.9	58.6	80.5	58.0	13.3
Sand (%; 0.05–2.0 mm)	22	21.2	37.8	69.5	38.7	12.2
Silt (%; 0.002–0.05 mm)	22	<0.1	1.1	3.6	1.3*	1.0
Clay (%; <0.002 mm)	22	<0.1	0.9	2.1	1.0	0.5

† Min denotes minimum value; Med denotes median value; Max denotes maximum value; Mean denotes the arithmetic average; SD denotes standard deviation; and \* denotes significant difference between fall 2003 and spring 2004 data using a paired  $t$ -test ( $P < 0.05$ ).

‡ DO, dissolved oxygen; SRP, soluble reactive P; TP, total P; EPC<sub>0</sub>, equilibrium P concentration.

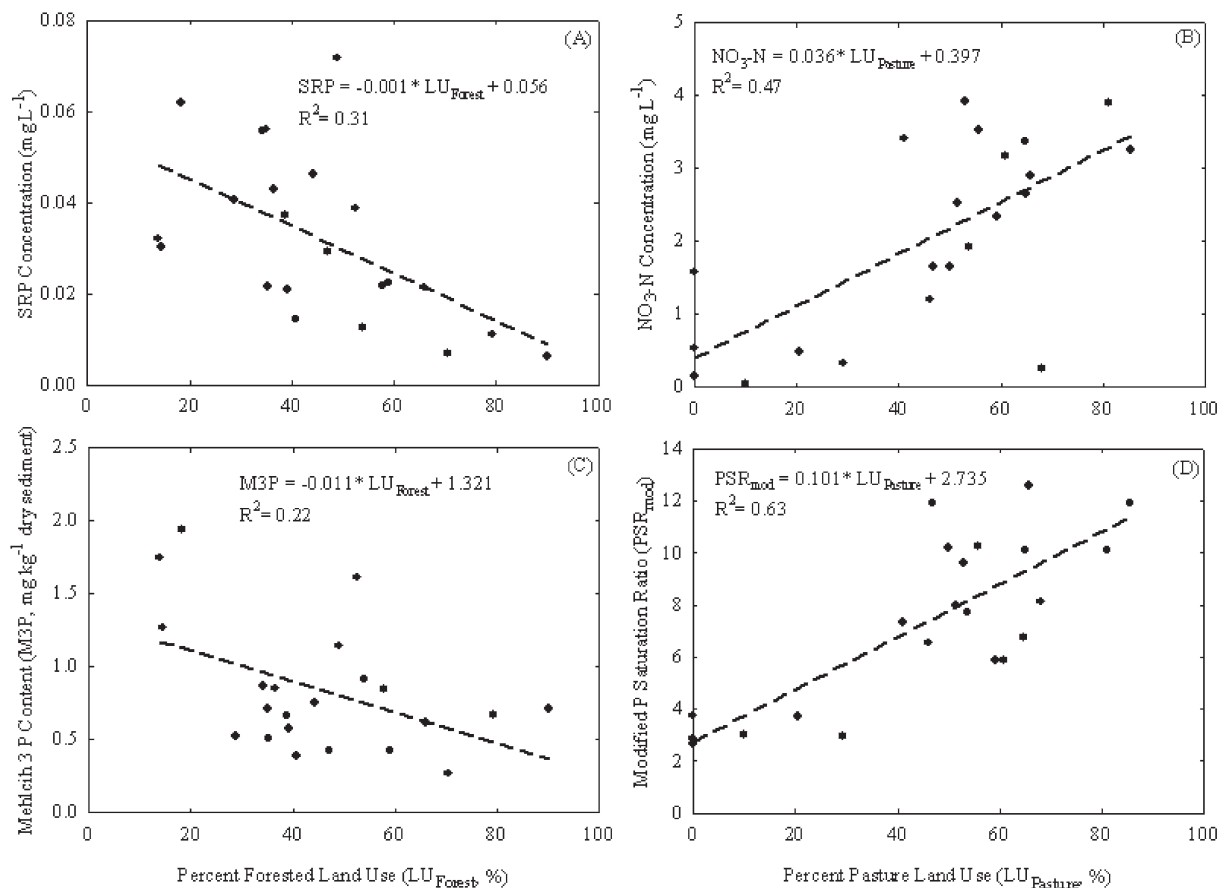


Fig. 3. The relation between (A) soluble reactive phosphorus (SRP) concentrations ( $\text{mg L}^{-1}$ ) in the water column and the percent (%) forested land use in the catchments; (B) nitrate-nitrogen ( $\text{NO}_3\text{-N}$ ) concentrations ( $\text{mg L}^{-1}$ ) in the water column at the selected stream and the percent (%) pasture land use in the catchments; (C) Mehlich-3 P (M3P) content ( $\text{mg kg}^{-1}$  dry sediment) in the stream sediments and the percent (%) forested land in the catchments; and (D) the modified P saturation ratio ( $\text{PSR}_{\text{mod}}$ ) of the stream sediments and the percent (%) pasture land use in the catchments.

category (ANOVA LSD,  $P < 0.05$ ) (Fig. 4A). Several studies have shown that agricultural land use (i.e., pasture) can influence water chemistry in ground and surface waters in the Ozark Highlands (Petersen et al., 1999; Haggard et al., 2003) and in other basins (e.g., see McFarland and Hauck, 1999; Buck et al., 2004). However, conductivity and Cl concentrations in the selected streams were positively correlated to the percent urban land use in the catchments (data not shown; Cond:  $r = 0.60$ ,  $P < 0.01$ ; Cl:  $r = 0.65$ ,  $P < 0.01$ ). The footprint of catchment land use on stream water chemistry, particularly nutrient concentrations, is rather consistent among studies, whether water quality monitoring was simply seasonal (as in this study) or more long term (multiple years as in the other citations).

### Sediment Equilibrium Phosphorus Concentrations

Benthic sediments displayed a linear P sorption isotherm (all regressions:  $R^2 \geq 0.75$ ,  $P \leq 0.05$ ), especially at initial aqueous SRP concentrations less than  $1.1 \text{ mg L}^{-1}$  (e.g., see Fig. 2). The linear portion of the sorption isotherm that was used to estimate sediment  $\text{EPC}_0$  often did not include the data from the highest level of P enrichment, i.e.,  $2.5 \text{ mg L}^{-1} \text{ PO}_4\text{-P}$  plus ambient SRP solution. This would suggest the possibility of a two-phase sorption process, and the isotherms from fall 2003 had a greater number of occur-

rences of this type than did isotherms from spring 2004. The slopes of the linear isotherms ( $K_{\text{slope}}$ ) were greater in fall 2003 compared to spring 2004 (paired  $t$ -test,  $P < 0.05$ ), which coincided with a greater proportion of silt particles (paired  $t$ -test,  $P < 0.001$ ) (Table 2 and 3). The slope of the linear isotherm ( $K_{\text{slope}}$ ) was positively correlated to the proportion of silt particles (0.002–0.05 mm) in the benthic sediments ( $r = 0.57$ ,  $P < 0.05$ ), suggesting that the P buffering capacity of these sediments increases with an increase in fine sediments. Overall, these benthic sediments were largely made up of particles sand size (0.05–2.0 mm) or greater (Table 2 and 3). Benthic sediment  $\text{EPC}_0$  also exponentially decreased ( $R^2 = 0.38$ ,  $P < 0.01$ ) with an increase in  $K_{\text{slope}}$ , which represents the ability of the sediments to adsorb P (Fig. 5D).

Sediment  $\text{EPC}_0$  varied by more than an order of magnitude (Table 2 and 3), ranging from less than  $0.001 \text{ mg L}^{-1}$  at Beaty Creek and Lollars Creek to  $0.329 \text{ mg L}^{-1}$  at Ballard Creek. The range in sediment  $\text{EPC}_0$  was much larger than that observed with average SRP concentration in the water column of the streams ( $0.003\text{--}0.072 \text{ mg L}^{-1}$ ). Sediment  $\text{EPC}_0$  at the selected streams was greater (paired  $t$ -test,  $P < 0.001$ ) than SRP concentrations in the water column, although sediment  $\text{EPC}_0$  and SRP concentrations were significantly correlated ( $r = 0.51$ ,  $P < 0.05$ ) across the selected streams (Fig. 5A). This observation suggests that benthic

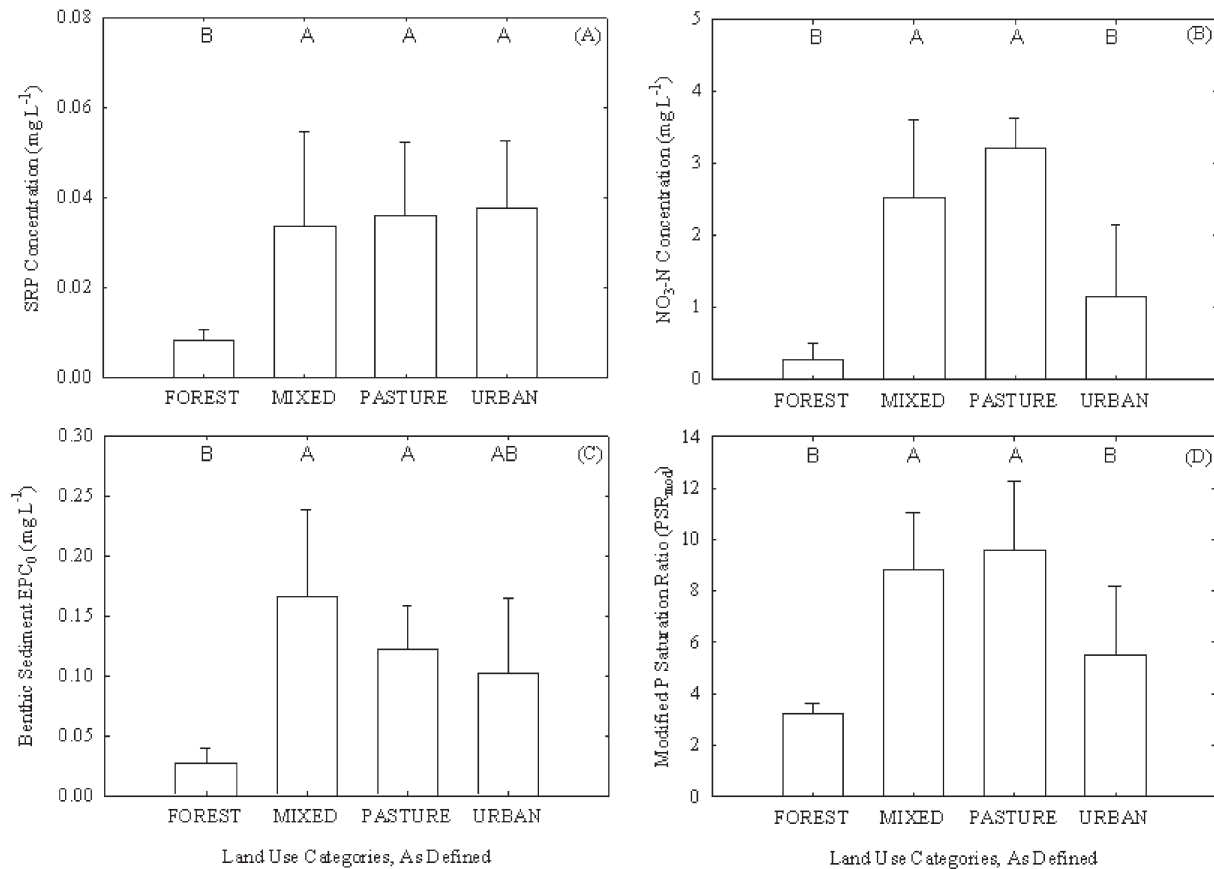


Fig. 4. Mean (plus standard deviation) of (A) soluble reactive phosphorus (SRP) concentrations ( $\text{mg L}^{-1}$ ), (B) nitrate-nitrogen ( $\text{NO}_3\text{-N}$ ) concentrations ( $\text{mg L}^{-1}$ ), (C) sediment equilibrium P concentrations ( $\text{EPC}_0$ ,  $\text{mg L}^{-1}$ ), and (D) the modified P saturation ratio ( $\text{PSR}_{\text{mod}}$ ) as a function of the various defined land use categories. Note: Different letters above bars denote significant differences between land use categories using ANOVA LSD  $P < 0.05$ .

sediments in Ozark streams play a role in the regulation and/or maintenance of SRP concentration in the water column, as observed in several other studies nationwide (e.g., see Klotz, 1988; McDowell et al., 2001; Novak et al., 2004). However, average sediment  $\text{EPC}_0$  explained only a small portion ( $R^2 = 0.26$ ) of the variability observed in average SRP concentrations, indicating that many other factors contribute to the regulation and maintenance of dissolved P in Ozark streams.

Benthic sediments might be a source of dissolved P to the overlying water in Ozark streams, because sediment  $\text{EPC}_0$  was greater than SRP and desorption of P often occurred in our lab studies at low initial SRP concentrations, i.e., generally  $0.1 \text{ mg PO}_4\text{-P L}^{-1}$  plus ambient SRP concentration or less. Sediment  $\text{EPC}_0$  was generally greater (ANOVA LSD,  $P < 0.05$ ) in the selected streams draining catchments in the *Pasture* or *Mixed* land use category (Fig. 4C). Benthic sediments in *Pasture* streams, or those draining catchments with a *Mixed* land use, may assist in maintaining elevated SRP concentrations. Other studies have also shown that benthic sediments adjacent to agricultural lands have a greater P content than those adjacent to forested lands (McDowell et al., 2002). However, municipal effluent discharges in Ozark streams have a much greater impact on sediment  $\text{EPC}_0$  than catchment land use, where sediment  $\text{EPC}_0$  downstream from effluent discharges has ranged from  $\sim 0.1$  to  $7 \text{ mg L}^{-1}$  (Popova et al., 2006; Ekka et

al., 2006). The selected streams in this study, including the highly urbanized streams, were not influenced from municipal effluent discharges, only nonpoint source impacts.

### Sediment Mehlich-3 Extractions

Contents of M3-extractable elements were highly variable between elements and within individual elements (Table 4). Contents of M3-extractable Al, Ca, Fe, K, Mg, Mn, and Na exceeded  $1 \text{ mg kg}^{-1}$  dry sediment at one or more of the selected sites, although M3-extractable elements were much less than the contents typically observed in soils (e.g., see Sims et al., 2002; Zhang et al., 2005; Brye, 2006). In particular, M3P contents in stream sediments are low ( $0.27\text{--}1.94 \text{ mg kg}^{-1}$  dry sediments) compared to levels considered optimal for productive agricultural soils ( $\sim 50 \text{ mg kg}^{-1}$ ) in Arkansas and other regions (e.g., Maguire and Sims, 2002). Contents of M3P in the sediments of these selected streams are much less than typical pasture soils in Arkansas, which range from less than  $10 \text{ mg kg}^{-1}$  in a soil never amended with poultry litter to more than  $400 \text{ mg kg}^{-1}$  in a soil that has received long-term applications of poultry litter (Daniels et al., 2001).

Several studies have evaluated the relation between soil M3P content and SRP concentrations in runoff water (Pote et al., 1999; Vadas et al., 2005), and our study showed that sediment M3P was correlated ( $r = 0.50$ ,  $P < 0.05$ ) to SRP concentrations in the stream

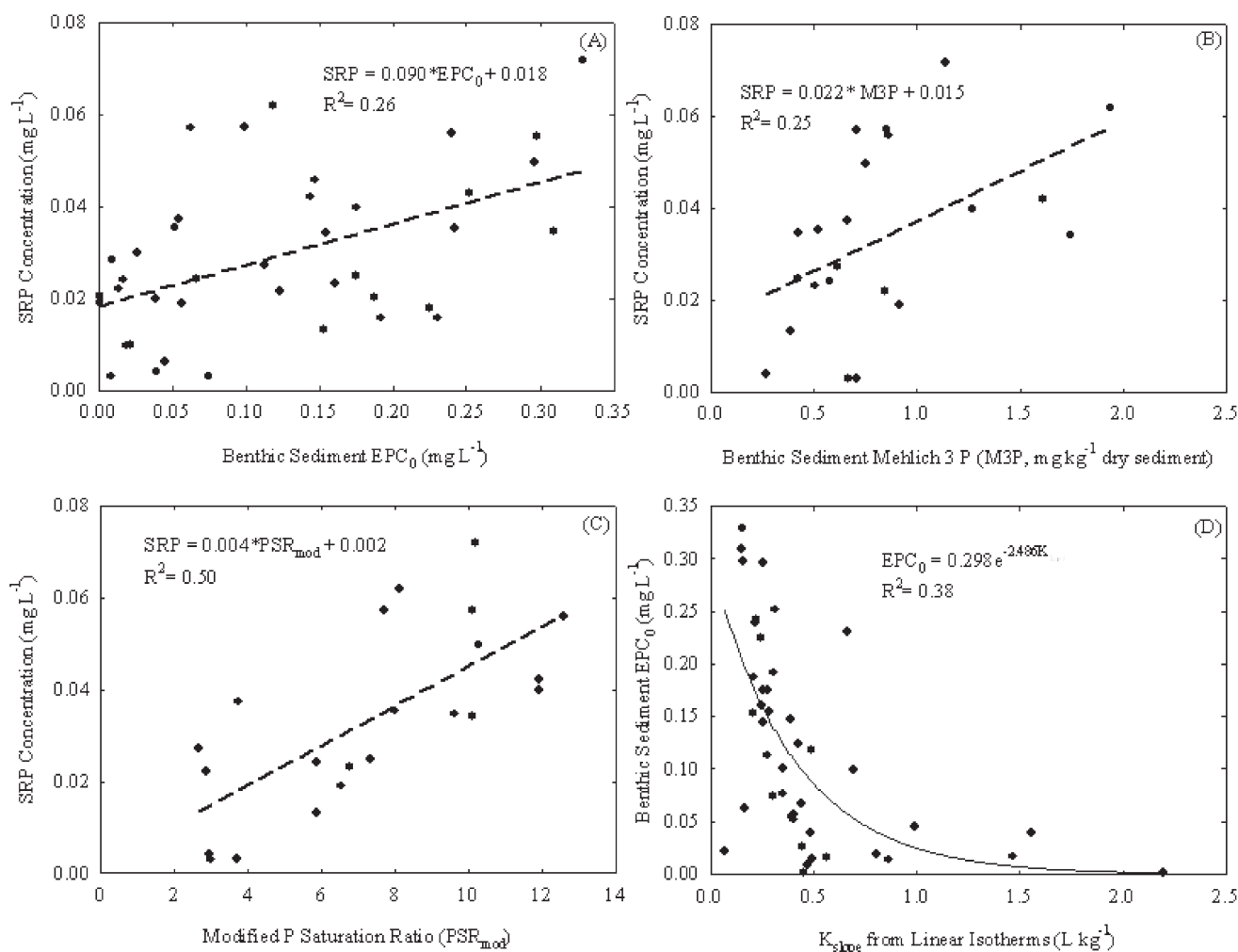


Fig. 5. The relation between (A) soluble reactive phosphorus (SRP) concentrations ( $\text{mg L}^{-1}$ ) in the water column and benthic sediment equilibrium P concentrations ( $\text{EPC}_0$ ,  $\text{mg L}^{-1}$ ) at the selected streams; (B) SRP concentrations ( $\text{mg L}^{-1}$ ) in the stream water and benthic sediment Mehlich-3 P (M3P) contents ( $\text{mg kg}^{-1}$  dry sediment); (C) SRP concentrations ( $\text{mg L}^{-1}$ ) in the stream water and the modified P saturation ratio ( $\text{PSR}_{\text{mod}}$ ) of the benthic sediments; and (D) benthic sediment  $\text{EPC}_0$  ( $\text{mg L}^{-1}$ ) and the slope ( $K_{\text{slope}}$ ,  $\text{L kg}^{-1}$ ) of the linear regression used to estimate benthic sediment  $\text{EPC}_0$ .

water (Fig. 5B). The slope of linear relation between SRP concentration and M3P content of the benthic sediments (0.022; Fig. 5B) is an order of magnitude greater than that observed by Vadas et al. (2005) between runoff SRP concentrations and M3P content of a wide range of soils (slope = 0.002). Even so, it suggests similar controls may exist between both solid and water phases in determining particulate-bound P release, and that sediment with low M3P contents (i.e., 0.2–2  $\text{mg M3P kg}^{-1}$  dry sediment) might release proportionately more dissolved P into aqueous solution compared to soils with greater M3P contents. Furthermore, sediment  $\text{EPC}_0$  and M3P content using step-wise linear regression explained about half of the variability observed in SRP concentrations at the selected streams ( $R^2 = 0.50$ ,  $\text{SRP} = 0.002 + 0.105 \text{EPC}_0 + 0.021 \text{M3P}$ ;  $P < 0.01$ ). Similar to SRP, M3P content in the sediments decreased ( $r = -0.47$ ,  $P < 0.05$ ) with an increase in the percent forested lands in the catchments (Fig. 3C).

Mehlich-3 extractions can be effectively used to evaluate P saturation in soils (Sims et al., 2002; Kleinman and Sharpley, 2002; Zhang et al., 2005), and PSRs are good indicators of the potential for P loss through various hydrologic pathways (Maguire and Sims,

2002; Allen et al., 2006). Mehlich-3 PSRs in soils generally range from less than 0.01 to as much as 0.59 (Sims et al., 2002; Zhang et al., 2005). However, M3 PSRs in the sediments of the selected Ozark streams were between 0.02 and 0.11 (Table 4) and on the lower end of the range typically observed in soils. McDowell and Sharpley (2001) observed that M3 PSRs ( $\sim 0.03$ , calculated from mean M3Al, M3Fe, and M3P) in bed sediments of an agricultural catchment were within the range observed in our study. Mehlich-3 PSRs of benthic sediments were not significantly correlated to sediment  $\text{EPC}_0$  or SRP concentration in the water column at the selected streams, as has been observed between M3 PSRs in soils and dissolved P concentrations in runoff water (Sims et al., 2002; Allen et al., 2006) or leachate (Maguire and Sims, 2002).

Mehlich-3 PSR and water-extractable P in calcareous soils have not shown a significant correlation (Ige et al., 2005), and other parameter combinations that included M3Ca and M3Mg provided a significant correlation. We used step-wise linear regression to determine what parameters were related to M3P and found that M3Fe, M3Mg, and  $K_{\text{slope}}$  of the linear sorption isotherm were significant factors ( $R^2 = 0.67$ ,  $\text{M3P} = 0.581 +$



0.084 M3Fe – 0.071 M3Mg – 0.977  $K_{slope}$ ,  $P < 0.05$ ); the contents of M3Fe and M3Mn were also highly correlated ( $r = 0.87$ ,  $P < 0.001$ ). The removal of M3Fe from the PSR denominator showed that M3P/M3Al in the sediments was not significantly correlated to SRP concentration in the stream water, whereas M3P/M3Fe was positively correlated to SRP concentration at these streams ( $r = 0.53$ ,  $P < 0.05$ ). However,  $PSR_{mod}$  was correlated ( $r = 0.71$ ,  $P < 0.001$ ) with SRP concentration in the water column at the selected streams (Fig. 5C), and substitution of M3Ca in place of M3Mg did not produce a significant correlation.

Some catchment-scale effect on the contents of M3-extractable elements and the degree of P saturation existed, similar to the effects observed on water chemistry and sediment  $EPC_0$ . The degree of P saturation (i.e.,  $PSR_{mod}$ ) in the sediments generally increased with an increase in the proportion of pasture land use in the catchments ( $r = 0.80$ ,  $P < 0.001$ ) (Fig. 3D). Sediments from the selected streams in the *Pasture* and *Mixed* land use catchments had greater  $PSR_{mod}$  compared to the other land use categories (ANOVA LSD,  $P < 0.01$ ) (Fig. 4D).

## Conclusions

This study evaluated the variation among water chemistry and sediment P interactions among streams draining catchments that varied in the percent forested, pasture, and urban land use (Objective 1). Soluble reactive P concentrations in the water column and M3P contents of the benthic sediments at the selected Ozark streams decreased with an increase in percent forested area in the catchments, whereas  $NO_3-N$  concentrations and  $PSR_{mod}$  increased with an increase in percent pasture land use within the catchments. Thus, catchment land use affects water chemistry and sediment P parameters measured in this study.

This study also evaluated the relations between SRP concentration, sediment  $EPC_0$ , and other measured abiotic factors in the stream sediments (Objective 2). Soluble reactive P concentrations in the water column of these selected streams were positively correlated to sediment  $EPC_0$ , although sediment  $EPC_0$  was generally greater than SRP at individual streams. However, sediment  $EPC_0$  explained only 26% of the variation in SRP concentrations, indicating that many other abiotic and/or biotic factors influence SRP.

This study evaluated the use of the traditional M3 extraction on stream sediments to elucidate other abiotic factors related to SRP concentration in stream sediments (Objective 3). Mehlich-3 P contents in benthic sediments were related to SRP concentration measured in the stream water, and the combination of M3P and sediment  $EPC_0$  explained over 50% of the variability in SRP concentrations. Mehlich-3 extractions were useful in estimating PSRs, and a modified PSR ( $PSR_{mod}$ ) of the benthic sediments was strongly correlated to SRP concentrations in the water column of the selected Ozark streams. Thus, M3P and  $EPC_0$  estimates from stream sediments may be valuable yet simple indicators of whether benthic sediments act as sinks or sources of P in fluvial systems, as well as estimating changes in stream SRP concentrations.

Table 4. Phosphorus saturation ratios ( $PSR$  and  $PSR_{mod}$ ) and contents of Mehlich 3 (M3)-extractable elements measured using inductively coupled plasma-optical emission spectrometry (ICP-OES) across the 22 selected sampling sites at Ozark streams in spring 2004 (May through June).

Element	n	Min†	Med	Max	Mean	SD
		mg kg <sup>-1</sup>				
M3Al	22	1.23	11.3	18.1	10.4	4.83
M3As	22	0.01	0.05	0.09	0.05	0.02
M3B	22	0.07	0.11	0.22	0.11	0.03
M3Ca	22	22.6	58.7	400	104	110
M3Cd	22	<0.01	<0.01	<0.01	<0.01	<0.01
M3Co	22	0.01	0.03	0.09	0.03	0.02
M3Cr	22	0.02	0.04	0.07	0.04	0.01
M3Cu	22	0.03	0.10	0.15	0.10	0.03
M3Fe	22	2.51	9.46	24.4	9.53	5.64
M3K	22	1.08	1.95	3.18	1.97	0.43
M3Mg	22	0.72	1.80	10.4	2.99	2.55
M3Mn	22	3.38	5.48	16.9	6.69	3.58
M3Mo	22	<0.01	<0.01	0.01	<0.01	<0.01
M3Na	22	29.3	32.8	34.7	32.4	1.64
M3Ni	22	<0.01	0.03	0.06	0.03	0.02
M3P	22	0.27	0.71	1.94	0.84	0.45
M3Pb	22	<0.01	0.06	0.20	0.06	0.06
M3S	22	0.45	0.57	2.66	0.90	0.65
M3Se	22	<0.01	0.01	0.03	0.01	0.01
M3Ti	22	0.01	0.04	0.12	0.04	0.03
M3Zn	22	0.15	0.25	0.99	0.34	0.22
PSR (molar basis)	22	0.023	0.047	0.108	0.051	0.022
$PSR_{mod}$ (molar basis)	22	0.027	0.075	0.126	0.074	0.032

† Min denotes minimum value; Med denotes median value; Max denotes maximum value; Mean denotes the arithmetic average; and SD denotes standard deviation.

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