



# Contrasting controls on the phosphorus concentration of suspended particulate matter under baseflow and storm event conditions in agricultural headwater streams



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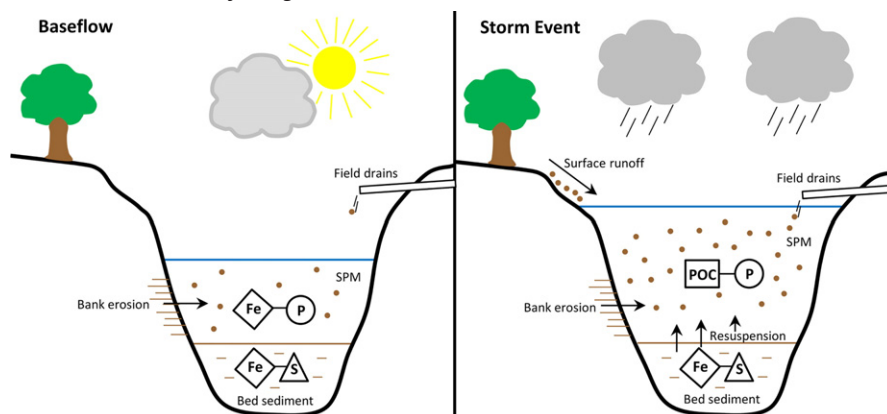
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## HIGHLIGHTS

- Controls on SPM P concentration are examined under differing stream flows.
- SPM P is dominantly associated with Fe under baseflow conditions.
- SPM P is dominantly associated with POC during storm events.
- Seasonality in the Fe–P and Al<sub>ox</sub>–Al<sub>di</sub> ratios of SPM are demonstrated.
- Changes in the sources of SPM are hypothesised as the causal mechanism.

## GRAPHICAL ABSTRACT

Conceptual diagram of the contrasting controls on SPM phosphorus concentration in agricultural headwater streams under different hydrological conditions.



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## ABSTRACT

Whilst the processes involved in the cycling of dissolved phosphorus (P) in rivers have been extensively studied, less is known about the mechanisms controlling particulate P concentrations during small and large flows. This deficiency is addressed through an analysis of large numbers of suspended particulate matter (SPM) samples collected under baseflow ( $n = 222$ ) and storm event ( $n = 721$ ) conditions over a 23-month period across three agricultural headwater catchments of the River Wensum, UK. Relationships between clay mineral and metal oxyhydroxide associated elements were assessed and multiple linear regression models for the prediction of SPM P concentration under baseflow and storm event conditions were formulated. These models, which explained 71–96% of the variation in SPM P concentration, revealed a pronounced shift in P association from iron (Fe) dominated during baseflow conditions to particulate organic carbon (POC) dominated during storm events. It is hypothesised this pronounced transition in P control mechanism, which is consistent across the three study catchments, is driven by changes in SPM source area under differing hydrological conditions. In particular, changes in SPM Fe–P ratios between small and large flows suggest there are three distinct sources of SPM Fe; surface

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soils, subsurface sediments and streambed iron sulphide. Further examination of weekly baseflow data also revealed seasonality in the Fe–P and aluminium oxalate–dithionate ( $Al_{ox}-Al_{di}$ ) ratios of SPM, indicating temporal variability in sediment P sorption capacity. The results presented here significantly enhance our understanding of SPM P associations with soil derived organic and inorganic fractions under different flow regimes and has implications for the mitigation of P originating from different sources in agricultural catchments.

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## 1. Introduction

Diffuse phosphorus (P) pollution is a key factor behind the development of eutrophic conditions in agricultural catchments (Withers and Jarvie, 2008; Quinton et al., 2010). As a naturally limiting nutrient of plant growth in aquatic environments, dissolved P (DP) enrichment fuels blooms of phytoplankton, periphyton and neuro-toxin secreting cyanobacteria colonies, which can dramatically lower species diversity and lead to a fundamental breakdown of ecosystem functioning (Smith et al., 1999; Hilton et al., 2006). Phosphorus is dominantly transported through rivers in particulate form, with sediment-associated P variously estimated to account for up to 90% of total P (TP) load in rural UK catchments (e.g. Walling et al., 1997; Bowes et al., 2003). However, there exists a dynamic equilibrium between the quantity of labile P associated with mineral surfaces and the concentration of DP in both soil solution (Hartikainen et al., 2010) and in stream water (Palmer-Felgate et al., 2009) which is controlled by biogeochemical processes. Consequently, understanding the importance of sediment biogeochemistry in controlling particulate P (PP) concentrations is essential if DP enrichment is to be mitigated.

Previous research has shown that DP reacts strongly with iron (Fe) and aluminium (Al) oxyhydroxide complexes in soils and stream sediments to form mineral-bearing PP phases. This occurs principally through the adsorption of phosphate ions onto solid phase mineral surfaces (non-occluded-P), followed by the subsequent absorption of phosphate ions into the mineral itself (occluded-P) (Walker and Syers, 1976; House and Denison, 2002; Evans et al., 2004). The rate at which this sorption process occurs is a function of the availability of potential P binding sites on particulate surfaces. This in turn is determined by factors such as mineral surface ionisation, presence of organic matter (OM) complexes and competition from other anions. Additionally, oxyhydroxides commonly bind to clay mineral surfaces via ligand exchange thereby forming an indirect association between P and the clay mineral component of soils and sediments (House and Warwick, 1999; Withers and Jarvie, 2008; Palmer-Felgate et al., 2009).

Although much is known about how these processes affect the instream cycling of P, less is known about how the relationships between PP and other organic and geochemical constituents change during small and large flow periods. This is important because previous research has demonstrated evidence of strong clockwise hysteresis in concentrations of both suspended particulate matter (SPM) and PP during storm events in agricultural headwater catchments (Stutter et al., 2008), thereby providing evidence of changing SPM P associations under varying flow conditions. Whilst studies by Van der Perk et al. (2007) and Rawlins (2011) developed regression models to demonstrate the importance of a range of elements (Al, Ca, Ce, Fe, K, Mn) and phases (Al/Fe oxyhydroxides, clay minerals, OM) in determining the P concentration of streambed sediments under baseflow conditions, neither study considered how these associations changed under differing flows. In fact, to our knowledge, no previous study has examined the geochemical associations between SPM and its P bearing phases under different hydrological conditions in agricultural headwater catchments. This represents a significant deficiency because source apportionment studies have demonstrated that there can be a significant change in the sources of SPM under differing flows, with subsurface inputs linked to baseflow sediment supply and increased surface source contributions associated with precipitation events (Cooper et al., 2015). Therefore, if

the sources of SPM change under different flow regimes, one can hypothesise that the organic and geochemical relationships between SPM and its P component may be similarly affected.

Therefore, the main objectives of this study were:

- (i) to compare and contrast SPM organo-mineral relationships under baseflow and storm event conditions;
- (ii) to develop statistical models to identify the importance of various organic and inorganic parameters in determining SPM P concentration under differing flow regimes;
- (iii) to explore evidence of seasonality in SPM P concentration, Fe–P ratios and Al/Fe oxalate–dithionate ratios, and to consider what these temporal trends reveal about variability in the sources of SPM P.

This study was conducted over a 23-month period in a tributary of the lowland River Wensum, UK, and formed part of a wider investigation into the sources of fine grained SPM in this agricultural catchment.

## 2. Methods

### 2.1. Study location

This study focused on the 20 km<sup>2</sup> Blackwater sub-catchment of the River Wensum, Norfolk, UK (Fig. 1). This intensive arable headwater catchment is monitored as part of the River Wensum Demonstration Test Catchment (DTC) project which aims to evaluate the extent to which on-farm mitigation measures can cost-effectively reduce diffuse agricultural pollution whilst maintaining food production capacity (Outram et al., 2014). The Blackwater sub-catchment is divided into six 'mini-catchments' A to F for observational purposes. Each mini-catchment has a bankside kiosk at the outlet monitoring a variety of water quality parameters (e.g. pH, turbidity, temperature, ammonium, chlorophyll, dissolved oxygen, stage) at 30-min resolution. Each kiosk also encompasses an automatic water sampler (Teledyne ISCO, Lincoln, NE) containing 24, 1 L polypropylene bottles which were activated during heavy precipitation events to sample stream water.

Here, we focused solely upon mini-catchments A (5.4 km<sup>2</sup>), B (1.3 km<sup>2</sup>) and E (7.12 km<sup>2</sup>), where mini-catchments A and B are nested within mini-catchment E. Situated 30–60 m above sea level with gentle slopes (<0.5°), intensive arable land constitutes 89% of these three headwater catchments, with 8% grassland, 2% mixed woodland and 1% rural settlements. The bedrock is Cretaceous white chalk at a depth of ~20 m. Overlaying this are superficial deposits of Mid-Pleistocene diamicton glacial tills, principally chalky, flint-rich boulder clays of the Sheringham Cliffs (0.2–0.5 m depth) and Lowestoft Formations (0.5–20 m depth), interspersed with layers of glaciofluvial and glaciolacustrine sands and gravels. Superimposed on this are deposits of Late Pleistocene silty loess (cover loam) and Holocene-age alluvium and river terrace material. The principal surface soil types are clay loam to sandy clay loam to a depth of >0.2 m (Hiscock et al., 1996; Lewis, 2011). A weather station at the outlet to mini-catchment A recorded average precipitation totals of 808 mm y<sup>-1</sup> and mean average annual temperatures of 9.2 °C during the April 2012–March 2014 monitoring period.

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