

# Efficacy of selected phosphorus sorbing materials (PSMs) to enhance the orthophosphate sorption capacity of filter socks

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## Keywords

diffuse pollution; filter socks; orthophosphate; phosphorus sorbing materials (PSMs); Water Framework Directive.

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## Abstract

Agricultural phosphate ( $\text{PO}_4^{3-}$ ) is a major cause of water quality compliance failures. Filter socks (FS) are a sediment-control method proven to be effective at removing sediment and sediment bound-P. Within the water and wastewater treatment industries phosphorous sorbing materials (PSMs) are widely used to remove reactive  $\text{PO}_4^{3-}$ . Combining PSMs with FS provides an opportunity to concomitantly remove sediment, sediment bound-P and  $\text{PO}_4^{3-}$  from agricultural runoff. A column experiment was undertaken to evaluate the efficacy of selected PSMs to remove  $\text{PO}_4^{3-}$  across a range of  $\text{PO}_4^{3-}$  concentrations (0.08, 0.26 and 1.3 mg/L) and contrasting FS fill media (PAS 100 Compost and Woodchip). The results indicate that Ca-enriched ferrihydrite removed up to 99% of  $\text{PO}_4^{3-}$ , and was the only PSM to reduce  $\text{PO}_4^{3-}$  to below the target value of <0.05 mg/L. An initial cost-benefit analysis indicates that PSM-enhanced FS are a viable option to remove  $\text{PO}_4^{3-}$  from agricultural runoff.

## Introduction

In England, only 83% of eligible waterbodies are currently meeting their EU Water Framework Directive (WFD) targets (Environment Agency, 2015). Agricultural diffuse pollution contributes 70% of sediments, 60% of nitrates ( $\text{NO}_3^-$ ) and 25% of phosphates ( $\text{PO}_4^{3-}$ ) to receiving waters (National Audit Office, 2010). This has resulted in increased ecological degradation through eutrophication, flooding, sedimentation and turbidity within the watercourses (Tilman *et al.*, 2002).

Phosphate losses from agricultural land are increasing due to the increased frequency of extreme rainfall events, and as farmers continue to inappropriately apply P-fertilisers (Tilman *et al.*, 2002; Schoumans *et al.*, 2014). Sediment-bound P can be managed through the use of erosion and sediment control measures, such as cover cropping, tillage management, buffer strips, filter socks (FS), grassed waterways and contour cropping (Faucette *et al.*, 2008; Shipitalo *et al.*, 2010; De Baets *et al.*, 2011; Vinten *et al.*, 2014; Mekonnen *et al.*, 2015; Mhazo *et al.*, 2016; Pan *et al.*, 2017; Zhang *et al.*, 2017).

However, methods to mitigate orthophosphate losses associated with agricultural runoff in-field are limited and include lime/alum application, reducing  $\text{PO}_4^{3-}$  inputs, establishment of hedges and shelterbelts, buffer strips

and land-use change (Deasy *et al.*, 2008; Newell Price *et al.*, 2011; Schoumans *et al.*, 2014). Consequently, the majority of elevated  $\text{PO}_4^{3-}$  in waterbodies is remediated by water treatment works (Defra, 2012). Removal typically involves filtration, precipitation and adsorption using metal ions (Yeoman *et al.*, 1988; De-Bashan and Bashan, 2004; Nancharaiah *et al.*, 2016). Phosphorus sorbing materials (PSMs) have been successfully utilised outside of the water treatment industry (Penn *et al.*, 2017) in the form of surface runoff confined bed filters and surface and subsurface tile drain P removal structures. PSM when combined with filter socks could also prove incredibly beneficial to the agricultural sector for in-field  $\text{PO}_4^{3-}$  control.

Commonly used PSMs include synthesised alumina (Mekhmer *et al.*, 2000; Karim *et al.*, 2011), synthesised ferrihydrite (Michel *et al.*, 2010; Wang *et al.*, 2013), synthesised goethite (Torrent *et al.*, 1990; Rakovan *et al.*, 1999), natural or synthesised calcite (Suzuki *et al.*, 1986; Li *et al.*, 2017), and clays, such as montmorillonite and illite (Gimsing and Borggaard, 2002; Ooi *et al.*, 2017). Additionally, PSMs are widely used in on-site wastewater treatment systems, such as infiltration beds, constructed wetlands, filterwells and tile drain P removal structures (Cucarella and Renman, 2009; Penn *et al.*, 2017). These PSMs, though researched are not widely utilised outside

of the water treatment industry, but could prove incredibly beneficial to the agricultural sector for in-field control of  $\text{PO}_4^{3-}$ .

To be compliant with WFD targets, the removal of sediment, sediment bound-P and  $\text{PO}_4^{3-}$  is necessary from the UK agricultural sector. This is in line with Rules 1 and 6 of the Farming Rules for Water, introduced in the United Kingdom in 2018; namely 'Pollution Risk' and 'Preventing Erosion' (GOV.UK, 2018). Filter socks have been found to effectively remove significant amounts of sediment and associated sediment bound-P from runoff (Faucette *et al.*, 2008, 2009; Cooke *et al.*, 2017). Filter socks are tubular geotextiles that are typically filled with compost or woodchip and are placed in-field at prescribed intervals, upslope of riparian buffers or in concentrated flow paths to intercept runoff. Filter socks work by:

- (a) acting as a temporary bund, allowing the runoff to initially pool upslope whereby sand and silt particles deposit; and
- (b) reducing the velocity of runoff as it passes through the filter sock, allowing the silt and clay particles to deposit within the filter sock fill media.

However, filter socks are currently not effective at removing  $\text{PO}_4^{3-}$ . Combining PSMs with filter socks provides an opportunity to concomitantly remove sediment, sediment bound-P and  $\text{PO}_4^{3-}$  from agricultural runoff.

The objective of this initial study was to critically evaluate the potential efficiency of natural, and by-product PSMs to remove  $\text{PO}_4^{3-}$  from runoff, in order to meet a pre-defined water quality target of 0.05 mg/L. This target was chosen as it is representative of the targets of many UK waterbodies, including the Lugg case study catchment (Cooke *et al.*, 2017). Designated waterbodies (e.g. Special Areas of Conservation) have  $\text{PO}_4^{3-}$  targets which are particularly stringent (<1 mg/L) to enable the habitats or species for which they are designated to be effectively protected. However, with a short time frame, and a lack of funding and appropriate knowledge of the catchments being operated in, the relevant authorities are struggling to meet these targets.

PSMs were selected based on their potential phosphate sorption capacity as indicated by the literature, their availability, and their potential cost with regards to future adoption.

The selected PSMs were mixed with typical filter sock fill media namely PAS 100 QP Compliant compost and woodchip. These were placed within a series of column experiments, whereby the PSM and fill media were exposed to a range of  $\text{PO}_4^{3-}$  solutions of known concentration. Volume of  $\text{PO}_4^{3-}$  solutions applied, mass of P applied and retention time were recorded to facilitate the determination

of PSE. Column experiments were chosen as they allowed mixing of the PSMs with the fill media, and due to their comparability with real-life conditions as compared with batch experiments (Helfferich, 1995; Loganathan *et al.*, 2014).

## Materials and methods

### Materials

Compost and woodchip were used in this study as the main filter sock 'fill media' into which the selected PSMs were mixed. This was to evaluate whether the PSM could effectively sorb  $\text{PO}_4^{3-}$  from both the applied  $\text{PO}_4^{3-}$  solution as well as and critically, sorb  $\text{PO}_4^{3-}$  released from the compost and woodchip fill media during 'flush events' (Faucette *et al.*, 2008). The study was not intended to replicate flow through a filter sock rather, the results would be used to identify potential PSMs that could then be applied within a filter sock context.

Synthesised Fe and Al-based PSMs are widely utilised in the water industry with  $\text{PO}_4^{3-}$  sorption occurring through ion and ligand exchange (mononuclear and binuclear bidentate coordination bonds), and surface precipitation (Ippolito *et al.*, 2011; Loganathan *et al.*, 2014; Gérard, 2016). Sorption is most effective at lower pH values,  $\text{pH} < 5$ , (Hingston *et al.*, 1968; Cornell and Schwertmann, 2003), and consequently PSMs that perform better at higher pH values, such as Ca and Mg, are sometimes used in conjunction to maximise  $\text{PO}_4^{3-}$  removal. However, synthesised materials are currently not suitable for widespread use in the agricultural sector for  $\text{PO}_4^{3-}$  control due to their comparatively high cost and inaccessibility as compared to waste and by-product PSMs. Consequently, the PSMs investigated in this study were natural ferrihydrite-rich ochre ( $\text{F}_{\text{Ca}}\text{-O}$ ) and goethite-rich ochre ( $\text{G-O}$ ), kaolin, fullers earth and a commercially available proprietary product. They were selected based on their availability and applicability to the UK agricultural sector, as well as their well-documented phosphate sorption capacity.

Detailed physico-chemical properties of the compost and woodchip fill media and the PSMs were established through standard laboratory and mineralogical analysis (Table 1). The compost and woodchip used within the treatments were not air dried to avoid them becoming hydrophobic, and were not wetted up, so that the target moisture content of <60% was maintained (Alexander Associates Inc., 2006). The compost was hand sieved prior to the experiment to meet the target particle size distribution, which maintains runoff flow-through rates of 15-38 L/min, whilst promoting sediment deposition (Alexander Associates, 2006). Similar specifications for

**Table 1** Baseline physico-chemical properties of the compost and woodchip fill media and PSMs

Property	Phosphorus Sorbing Material (PSM)						
	C	W	Pp	K	FE	F <sub>Ca</sub> -O	G-O
Particle size distribution (%)							
<0.002 mm	0	0	4.3	99	66	2.3	40.1
0.002–2 mm	0	0	95.7	1	34	97.7	59.9
<2 mm	63	57	0	0	0	0	0
2–5 mm	24	33	0	0	0	0	0
5–10 mm	12	10	0	0	0	0	0
>10 mm	1	1	0	0	0	0	0
Total P (mg/kg)	179 (±4.6)	148 (±5.0)	-	-	-	-	-
Particle density (g/cm <sup>3</sup> )	1.42	1.4	2.13	2.25	2.63	3.28	4.03
Specific surface area (m <sup>2</sup> /kg)	-	-	-	3213	730	588	945
Cation exchange capacity (cmol/kg)	-	-	13.5	2.9	64	0	0
Bulk mineralogy (%)							
Anatase	-	-	0	0	0.2	0	0
Aragonite	-	-	0.1	0	0	2.3	0
Calcite	-	-	0	0.6	2.0	50.4	14.1
Clinoptilolite	-	-	65.8	0	0	0	0
Ferrihydrite	-	-	0	0	0	46.6	0
Goethite	-	-	0.5	0	0	0	84.7
Hematite	-	-	0	0	0.7	0	0
Kaolinite	-	-	0	95.5	0	0	0
K-feldspar	-	-	0.1	0	21.0	0	0
Mica	-	-	0	0	2.0	0	0
Muscovite	-	-	0	3.9	0	0	0
Opal-C/CT	-	-	19.0	0	0	0	0
Plagioclase	-	-	0	0	8.2	0	0
Quartz	-	-	1.0	0	2.0	0.7	1.2
Smectite	-	-	13.5	0	64.0	0	0

'-' denotes where the analysis was not available for a particular material.

C, PAS 100 QP compliant compost; W, woodchip; Pp, proprietary product; K, kaolinite; FE, fullers earth; F<sub>Ca</sub>-O, ferrihydrite-ochre; G-O, goethite-ochre.

woodchip are not available, however, hand sieving was also undertaken to ensure a particle size distribution comparable to that of compost.

### Column trial methodology

The bulk mineralogy of the PSMs indicates that the proprietary product, kaolinite and Fullers Earth (are dominated by, clinoptilolite (65.8%), kaolinite (95.5%) and smectite (64.0%), respectively (Table 1). The calcium-enriched ferrihydrite ochre (F<sub>Ca</sub>-O) consists of 46.6% ferrihydrite and 50.4% calcite a stable polymorph of calcium carbonate. In contrast, the goethite ochre is dominated by goethite (84.7%) with 14.1% calcite (Table 1). Specific surface area is in the order kaolinite > G-O > Fullers Earth > F<sub>Ca</sub>-O with values of 3213, 945, 730 and 588 m<sup>2</sup>/kg, respectively. All PSMs were air-dried and mixed thoroughly with the compost or woodchip fill media at the proprietary product application rate (20 kg/m<sup>3</sup>). The fill media-PSM mixtures were packed into columns (10 cm length x 6.8 cm diameter PPE pipe

with the base covered by fabric gauze) to a bulk density of 700 kg/m<sup>3</sup>, replicating the desired bulk density of fill media in filter socks. Once packed, the columns were then clamped to a frame and leachate collected (Figure S1 of Supporting Information). Each column had between 182 and 191 ml of PO<sub>4</sub><sup>3-</sup> solution applied to achieve treatment saturation, based on the particle density and total porosity of the fill media-PSM mixtures (Table S1-2 of Supporting Information and Equations 1-4).

Fill media saturation (Supporting Information; Table S2) was achieved with deionised water (control), or the PO<sub>4</sub><sup>3-</sup> solution as a function of the treatment. The PO<sub>4</sub><sup>3-</sup> concentrations investigated in this study were selected based on values associated with runoff from the dominant erodible soils in the Lugg case study catchment (Cooke *et al.*, 2017):

- 'Low': 0.08 mg/L
- 'Medium': 0.26 mg/L
- 'High': 1.30 mg/L

These  $\text{PO}_4^{3-}$  concentrations are all above the water quality target for the Lugg catchment of  $<0.05$  mg/L, and were used to calculate the  $\text{PO}_4^{3-}$  load (as mg P) applied to the treatments (Supporting Information; Equation 4). This load was 0.015 mg P for the 'Low'  $\text{PO}_4^{3-}$  solution and ranged from 0.047 to 0.050 mg P and 0.237 to 0.251 mg P for the 'Medium' and 'High'  $\text{PO}_4^{3-}$  solutions (Supporting Information Table S3).

All treatments were replicated in quadruplicate. Two applications of  $\text{PO}_4^{3-}$  solution or deionised water (the control) were applied, 48 hours apart; Flush 1 and Flush 2, representing the mean time between rainfall events experienced in the Lugg catchment field trials (Cooke *et al.*, 2017). Solutions were evenly applied to the top of the columns, using the surface gauze to minimise preferential flow paths. The time taken for leachate initiation and leachate volume at 30 seconds, 1, 2, 5, 10, 20, 40, 60 mins postapplication was monitored, and ambient air temperature was recorded, as the temperature can influence PSM performance (Bai *et al.*, 2017). Leachate samples were filtered using  $< 0.45\mu\text{m}$  syringe filters prior to analysis. For Flush 1, between 57–63% and 34–63% of the volume of solution applied to the compost and woodchip PSM blended treatments was leached within 60 mins of solution application. This also indicates that between 37–43% and 37–66% of the volume of solution applied to the compost and woodchip PSM blended treatments were retained for 48hr prior to the application of Flush 2. Similar results were observed for Flush 2 (Supporting Information Table S4).

### Treatment performance

$\text{PO}_4^{3-}$  removed ( $PR$ ) by the PSM treatments was calculated using (Equation 5):

$$PR \text{ (mg)} = (s + t) - u \quad (5)$$

where  $s$  is the mean ( $n = 4$ ) leachate  $\text{PO}_4^{3-}$  load (mg) for the control (un-amended Compost and Woodchip) treatments where deionised water was applied;  $t$  is the mass of  $\text{PO}_4^{3-}$  applied to each treatment (Supporting Information Table S3);  $u$  is the leachate  $\text{PO}_4^{3-}$  load (mg).

Treatment performance was assessed by  $\text{PO}_4^{3-}$  sorption efficiency (PSE). Treatment PSE was determined using the mean ( $n = 4$ ) leachate  $\text{PO}_4^{3-}$  concentration for each of the treatments and the applied  $\text{PO}_4^{3-}$  load (Equation 6):

$$\text{Sorption efficiency (\%)} = \frac{(P0 + P1) - P2}{(P0 + P1)} \times 100 \quad (6)$$

T-tests (Statistica 13.2, Dell Inc.) compared F1 and F2 within treatment performance ( $p \leq 0.05$ ). One-way ANOVA and

*post hoc* Fisher LSD (Statistica 13.2 Dell Inc.) compared between treatment performance for each flush event (values followed by different letters are statistically different at  $p \leq 0.05$ ).

To facilitate the adoption of PSM-enhanced FS technology, and to assess the potential uptake of the PSMs within an agricultural context, an initial treatment cost-benefit was determined following (Equation 7).

$$PCM = \frac{1}{\left(\frac{PR}{Q+V}\right)} \times MC \quad (7)$$

where  $PCM$  is the  $\text{PO}_4^{3-}$  load removed per gram of column material.  $PR$  is  $\text{PO}_4^{3-}$  removal (Equation 5);  $Q$  is the mass of column fill media (254g); and  $V$  is the mass of PSM (0 g or 7.25 g).

Dividing by 1 gives the amount of material required for 1 mg P removal. Literature material costs ( $MC$ ) were averaged per gram of PSM material.

## Results

The leachate  $\text{PO}_4^{3-}$  concentration (Table 2) varied significantly between and within compost and woodchip treatments. FCa–O was the most successful PSM, regardless of fill media, and consistently met the water quality target of  $<0.05$  mg/L. However, for both compost and woodchip fill media, across all  $\text{PO}_4^{3-}$  applications and flushes, kaolinite, proprietary product and Fullers Earth PSMs were ineffective at reducing leachate  $\text{PO}_4^{3-}$  concentrations, compared to the non-PSM control (Table 2). This is reflected in the phosphate sorption efficiency (PSE) which was used to quantify treatment performance for both Flush 1 and Flush 2 (Tables 3 and 4).

### Compost treatments

#### Leachate concentrations

Flush 1 results indicate that for both the Low and High  $\text{PO}_4^{3-}$  applications, the leachate  $\text{PO}_4^{3-}$  concentrations of the compost FCa–O treatment were significantly lower compared to all other treatments. At the Flush 1 Medium  $\text{PO}_4^{3-}$  application, the compost FCa–O treatment was associated with lower leachate  $\text{PO}_4^{3-}$  concentrations than all other treatments except compost G–O. This indicates that Fe-oxides were the best performing PSMs when placed in compost fill media. Similar results were observed for Flush 2 (Table 2). Furthermore, for the Flush 2 High  $\text{PO}_4^{3-}$  applications, the compost G–O and compost FCa–O treatment leachate  $\text{PO}_4^{3-}$  concentrations were significantly lower than the other treatments.

**Table 2** Differences in mean  $\text{PO}_4^{3-}$  concentration ( $\text{mg P L}^{-1}$ ) for each PSM treatment at F1 and F2 as compared to the non-PSM Compost (C) and Woodchip (W) controls

Compost Fill media							
$\text{PO}_4^{3-}$ concentration <sup>1</sup>	Flush	Non-PSM control (C)	CPp	CK	CFE	CFCa–O	CG–O
Low	F1	1.13 <sup>c</sup>	0.93 <sup>c</sup>	1.22 <sup>c</sup>	1.02 <sup>c</sup>	0.01 <sup>a</sup>	0.48 <sup>b</sup>
	F2	1.73 <sup>d</sup>	1.60 <sup>d</sup>	1.48 <sup>cd</sup>	1.10 <sup>bc</sup>	0.43 <sup>a</sup>	0.76 <sup>ab</sup>
Medium	F1	1.05 <sup>b</sup>	1.03 <sup>b</sup>	1.18 <sup>b</sup>	1.12 <sup>b</sup>	0.04 <sup>a</sup>	0.36 <sup>a</sup>
	F2	2.15 <sup>d</sup>	1.38 <sup>bc</sup>	1.73 <sup>cd</sup>	1.38 <sup>bc</sup>	0.22 <sup>a</sup>	0.82 <sup>ab</sup>
High	F1	1.47 <sup>c</sup>	1.73 <sup>c</sup>	2.00 <sup>c</sup>	1.68 <sup>c</sup>	0.04 <sup>a</sup>	0.90 <sup>b</sup>
	F2	2.40 <sup>c</sup>	2.20 <sup>c</sup>	1.98 <sup>c</sup>	2.03 <sup>c</sup>	0.49 <sup>a</sup>	1.08 <sup>b</sup>
Woodchip Fill media							
$\text{PO}_4^{3-}$ concentration <sup>1</sup>	Flush	Non-PSM control (W)	WPp	WK	WFE	WFCa–O	WG–O
Low	F1	14.2 <sup>bc</sup>	19.6 <sup>c</sup>	15.1 <sup>bc</sup>	14.5 <sup>bc</sup>	9.48 <sup>b</sup>	1.15 <sup>a</sup>
	F2	40.0 <sup>b</sup>	44.1 <sup>b</sup>	40.5 <sup>b</sup>	37.9 <sup>b</sup>	0.55 <sup>a</sup>	10.2 <sup>a</sup>
Medium	F1	17.5 <sup>c</sup>	15.5 <sup>bc</sup>	19.9 <sup>c</sup>	11.5 <sup>abc</sup>	2.70 <sup>a</sup>	6.5 <sup>ab</sup>
	F2	47.0 <sup>bc</sup>	42.4 <sup>bc</sup>	47.9 <sup>c</sup>	33.0 <sup>b</sup>	0.81 <sup>a</sup>	9.48 <sup>a</sup>
High	F1	18.5 <sup>c</sup>	13.9 <sup>bc</sup>	25.1 <sup>d</sup>	9.68 <sup>b</sup>	0.75 <sup>a</sup>	9.23 <sup>b</sup>
	F2	52.8 <sup>c</sup>	47.8 <sup>bc</sup>	55.1 <sup>c</sup>	33.2 <sup>b</sup>	0.70 <sup>a</sup>	7.48 <sup>a</sup>

Water quality P target: 0.05 mg/L

Pp, proprietary product; K, kaolinite; FE, fullers earth; FCa–O, ferrihydrite-ochre; G–O, goethite-ochre.

<sup>1</sup>For each fill media, solution  $\text{PO}_4^{3-}$  concentration and Flush (F1 and F2) values in each row followed by the same letter are not significantly different following Factorial ANOVA and *post hoc* Fisher LSD analysis.

**Table 3** Differences in mean PSE (%) for each treatment and flush, compared to the control

Compost fill media							
$\text{PO}_4^{3-}$ load <sup>1</sup>	Flush	Non-PSM control (C)	CPp	CK	CFE	CFCa–O	CG–O
Low	F1	15.3 <sup>c</sup>	29.4 <sup>c</sup>	6.4 <sup>c</sup>	22.5 <sup>c</sup>	99.0 <sup>a</sup>	62.4 <sup>b</sup>
	F2	–47.6 <sup>d</sup>	–39.2 <sup>d</sup>	–29.0 <sup>cd</sup>	4.3 <sup>bc</sup>	61.6 <sup>a</sup>	31.8 <sup>ab</sup>
Medium	F1	30.5 <sup>c</sup>	31.0 <sup>c</sup>	20.8 <sup>c</sup>	24.8 <sup>c</sup>	97.1 <sup>a</sup>	75.2 <sup>b</sup>
	F2	–59.5 <sup>d</sup>	–3.3 <sup>bc</sup>	–30.2 <sup>cd</sup>	–3.3 <sup>bc</sup>	83.0 <sup>a</sup>	36.9 <sup>ab</sup>
High	F1	42.3 <sup>bc</sup>	31.8 <sup>c</sup>	20.7 <sup>c</sup>	33.8 <sup>c</sup>	98.3 <sup>a</sup>	64.1 <sup>b</sup>
	F2	–0.6 <sup>c</sup>	7.1 <sup>c</sup>	16.4 <sup>c</sup>	14.5 <sup>c</sup>	79.2 <sup>a</sup>	54.0 <sup>b</sup>
Woodchip fill media							
$\text{PO}_4^{3-}$ load <sup>1</sup>	Flush	Non-PSM control (W)	WPp	WK	WFE	WFCa–O	WG–O
Low	F1	28.6 <sup>bc</sup>	0.1 <sup>c</sup>	22.4 <sup>bc</sup>	25.9 <sup>bc</sup>	50.7 <sup>b</sup>	93.9 <sup>a</sup>
	F2	26.1 <sup>b</sup>	17.5 <sup>b</sup>	23.5 <sup>b</sup>	28.8 <sup>b</sup>	99.0 <sup>a</sup>	80.2
Medium	F1	12.5 <sup>c</sup>	21.7 <sup>bc</sup>	–1.2 <sup>c</sup>	41.9 <sup>abc</sup>	86.1 <sup>a</sup>	66.0 <sup>ab</sup>
	F2	13.4 <sup>bc</sup>	21.0 <sup>bc</sup>	9.9 <sup>c</sup>	38.2 <sup>b</sup>	98.5 <sup>a</sup>	81.7 <sup>a</sup>
High	F1	12.5 <sup>c</sup>	33.5 <sup>bc</sup>	–21.5 <sup>d</sup>	53.4 <sup>b</sup>	96.3 <sup>a</sup>	54.2 <sup>b</sup>
	F2	4.6 <sup>c</sup>	12.7 <sup>bc</sup>	–1.7 <sup>c</sup>	38.9 <sup>b</sup>	98.7 <sup>a</sup>	85.8 <sup>a</sup>

Pp, proprietary product; K, kaolinite; FE, fullers earth; FCa–O, ferrihydrite-ochre; G–O, goethite-ochre.

<sup>1</sup> $\text{PO}_4^{3-}$  load applied via 'Low-P' (0.08 mg/L); 'Medium-P' (0.26 mg/L); 'High-P' (1.3 mg/L) concentration  $\text{PO}_4^{3-}$  solutions. All results are compared to the PSE of the non-PSM C or W treatments. Therefore, negative values indicate an increase in leachate  $\text{PO}_4^{3-}$  compared to the control and consequently are recorded as negative PSE. Values within each row followed by the same letter are not significantly different following One-Way ANOVA and *post hoc* Fisher LSD analyses.

Significantly higher leachate  $\text{PO}_4^{3-}$  concentrations were found for Flush 2 than Flush 1, owing to the release of fill media  $\text{PO}_4^{3-}$  into the solution retained by the treatments

during the 48hr period between the Flush 1 and Flush 2 events. Additionally, some PSM adsorption sites will have been occupied during Flush 1.



**Table 4** T-test *p*-values indicating significant differences ( $p < 0.05$ ) in PSE between PSM treatments across Low, Medium and High solution  $\text{PO}_4^{3-}$  concentrations

Compost fill media							
Phosphate*	Flush	Non-PSM control (C)	CPp	CK	CFE	FCa–O	CG–O
Low	F1	0.00	0.00*	0.05*	0.19	0.16	0.06
	F2						
Medium	F1	0.07	0.03*	0.04*	0.13	0.28	0.01*
	F2						
High	F1	0.10	0.07	0.47	0.21	0.11	0.04*
	F2						
Woodchip fill media							
Phosphate*	Flush	Non-PSM control (W)	WPp	WK	WFE	WCa–O	WG–O
Low	F1	0.78	0.28	0.95	0.82	0.00*	0.04*
	F2						
Medium	F1	0.94	0.96	0.59	0.85	0.21	0.33
	F2						
High	F1	0.71	0.27	0.18	0.33	0.05*	0.00*
	F2						

Pp, proprietary product; K, kaolinite; FE, fullers earth; FCa–O, ferrihydrite-ochre; G–O, goethite-ochre.

\*T-test *p*-values are significantly different at a 95% confidence interval.

### PSM performance

The compost FCa–O treatment consistently recorded significantly higher PSE (Table 3) than all other treatments (except the compost G–O treatment at Flush 2 Low and Medium  $\text{PO}_4^{3-}$  applications). PSE for the compost FCa–O and compost G–O treatments ranged from 61.6 to 99.0% and 31.8 to 75.2%, respectively (Table 3). Additionally, no significant differences in PSE were observed between Flush 1 and Flush 2 (Table 4).

Though less consistent than the compost FCa–O treatment, the compost G–O treatment was repeatedly associated with significantly higher PSE, when compared to compost amended with proprietary product, kaolinite and Fullers Earth (Table 3). Furthermore, the compost G–O treatment demonstrates a significant reduction in PSE between Flush 1 and Flush 2 (Table 4), suggesting the partial saturation of PSM sorption sites.

Few significant differences in PSE were observed between the compost proprietary product, kaolinite, Fullers Earth amended treatments and un-amended compost control, and were associated with negative efficiency values (Table 3), indicating higher leachate  $\text{PO}_4^{3-}$ . This is likely due to the partial saturation of PSM sorption sites, and  $\text{PO}_4^{3-}$  desorption from the compost fill media.

Both the compost proprietary product and kaolinite amended treatments are considered ineffective, as they consistently demonstrate no significant difference in PSE as compared to the un-amended compost control. Significant differences between Flush 1 and Flush 2 for

the un-amended compost control, and proprietary product and kaolinite amended compost treatments (Table 4) are due to negative Flush 2 PSE, indicating that greater  $\text{PO}_4^{3-}$  is released from the fill media than is sorbed by the PSM.

### Woodchip treatments

#### Leachate concentrations

For the Flush 1 Low  $\text{PO}_4^{3-}$  application, the woodchip G–O treatment was associated with a significantly lower leachate  $\text{PO}_4^{3-}$  concentration, compared to all other treatments. For the Flush 1 Medium  $\text{PO}_4^{3-}$  application, leachate  $\text{PO}_4^{3-}$  concentration is in the order woodchip FCa–O = G–O = Fullers Earth < proprietary product = kaolinite = non-PSM woodchip control (Table 2). In contrast, at High  $\text{PO}_4^{3-}$  application, the woodchip FCa–O treatment was associated with significantly lower leachate  $\text{PO}_4^{3-}$  concentrations, compared to all other treatments.

For Flush 2, across all solution applications, the woodchip FCa–O and G–O treatments are associated with the lowest leachate  $\text{PO}_4^{3-}$  concentrations; however, the water quality target was not achieved (Table 2). This is due to the significantly higher leachate  $\text{PO}_4^{3-}$  concentrations associated with the W fill media. Following the addition of deionised water, mean un-amended compost and woodchip treatment leachate  $\text{PO}_4^{3-}$  concentrations for Flush 1 and Flush 2 were 1.3 and 19.8 and 1.1 and 54.0 mg/L, respectively (for brevity, data not shown).

## PSM performance

Except the Flush 1 Medium  $\text{PO}_4^{3-}$  application, PSE for woodchip FCa–O and G–O treatments is significantly higher than all other treatments (Table 3). Significant differences in PSE were observed for woodchip FCa–O and G–O treatments (Table 4) between Flush 1 and Flush 2. For the woodchip G–O treatment this is due to significantly lower PSE at Flush 2 than Flush 1. For the woodchip FCa–O treatment, Flush 2 PSE is significantly higher than that of Flush 1.

The PSE of woodchip treatments amended with proprietary product and Fuller Earth and the un-amended woodchip control is regularly significantly higher than the woodchip kaolinite treatments. No significant differences in PSE were observed between the Flush 1 and Flush 2 results for woodchip amended with proprietary product, Fullers Earth, kaolinite, or the un-amended woodchip control (Table 4).

## Discussion

### Efficacy of the PSMs to remove $\text{PO}_4^{3-}$

Across both fill media, FCa–O was the most efficient PSM at  $\text{PO}_4^{3-}$  removal, with PSE regularly exceeding 90% and leachate  $\text{PO}_4^{3-}$  concentrations of < 0.05 mg/L achieved on three occasions. This level of PSE could help to fail waterbodies realise their water quality targets.

The water quality target was not achieved by the G–O treatments, which had significantly lower PSE than FCa–O at Flush 1 (Low, Medium and High) and Flush 2 (High) when mixed with compost fill media, and at Flush 1 (High) when mixed with woodchip fill media. However, G–O still provided 31.8–75.2% PSE and may be preferable in locations where FCa–O is expensive and/or unavailable.

The FCa–O investigated in this study contains ferrihydrite (46.6%) and calcite (50.4%) (Table 1). Ferrihydrite is an amorphous Fe-oxide with two main configurations; 2-line and 6-line. FCa–O ferrihydrite is likely to be the 6-line form owing to its relatively large particle size (>0.002  $\mu\text{m}$ ; Table 1), resulting in fewer reactive surface sites (Arai and Sparks, 2001; Ballantine and Tanner, 2010; Gérard, 2016). With exceptionally high specific surface areas (SSAs); regularly up to 700  $\text{m}^2/\text{g}$  (Willet *et al.*, 1988; Wang *et al.*, 2013), ferrihydrite is able to provide large numbers of reactive surface sites, whereby  $\text{PO}_4^{3-}$  adsorption occurs through coordination and ionic bonding, creating monodentate or bidentate bonds (Parfitt, 1989; Wang *et al.*, 2013). The high calcium carbonate levels (Table 1) associated with FCa–O may allow it to sorb  $\text{PO}_4^{3-}$  from runoff with both low (ferrihydrite predominates adsorption) and higher (calcium carbonate predominates adsorption) pH values (Perassi and Borgnino, 2014; Xu *et al.*, 2014) increasing its applicability to a range of soil types and runoff pH levels.

$\text{PO}_4^{3-}$  adsorption by G–O is likely to occur through similar mechanisms to FCa–O (Tejedor-Tejedor and Anderson, 1990; Olsson *et al.*, 2010; Wei *et al.*, 2014). However, as G–O contains less calcium carbonate (calcite = 14.4%; Table 1), has a larger particle size, and smaller SSA (Table 1), less adsorption occurs than with FCa–O.

However, goethite is more commonly found than FCa–O in ochre deposits as it is more stable (Chesworth, 2008), and therefore may be more widely available.

The kaolinite, proprietary product and Fullers Earth PSMs were significantly less efficient at sorbing  $\text{PO}_4^{3-}$  than FCa–O and G–O, and had highly variable results, consistent with the literature (Gimsing and Borggaard, 2002; Gérard, 2016). The kaolinite treatments regularly exhibited negative PSE results. Similar results were observed by Krumina *et al.* (2016) who indicated negative values may originate from the saturation of the kaolinite with  $\text{PO}_4^{3-}$ , followed by removal of the labile fraction following additional  $\text{PO}_4^{3-}$  application. Furthermore, during the experiment, fine (<0.002 mm) kaolinite particles were regularly observed leaching out of the columns, unlike that of all other PSMs, resulting in there being less kaolinite for  $\text{PO}_4^{3-}$  adsorption. This is likely due to the smaller particle size of the K (99% <0.002mm (Table 1)) which did not allow retention within the fill media.

The Fullers Earth treatments were more consistent across flushes and  $\text{PO}_4^{3-}$  applications with only one negative PSE result observed. Consistency of performance is attributed to its relatively large SSA (Table 1) as well as the presence of smectites, namely montmorillonite. These have a 2:1 layer structure with a weak bond between layer sheets, and an unbalanced charge, causing them to be more chemically reactive (Odom, 1984). Consequently, the Fuller Earth provides more reactive surface sites than kaolinite for  $\text{PO}_4^{3-}$  adsorption (Uddin, 2017).

The proprietary product is commercially available and used for the control of  $\text{PO}_4^{3-}$  in the runoff. When compared to the non-PSM control, the proprietary product treatments often provide statistically similar or significantly less  $\text{PO}_4^{3-}$  sorption. When compared to other PSMs, FCa–O and G–O provide highly significant order of magnitude increases in PSE. Kaolinite, Fullers Earth and the proprietary product were all associated with variable results especially when compost was used as the fill media.

### Fill media

Generally, the results indicate a more consistent PSE from the woodchip, compared to compost fill media PSM treatments, as there were less negative PSE results reported. This is likely due to the longer retention time of the  $\text{PO}_4^{3-}$  solution within the woodchip fill media which retained, on average, 41% more of the solution in Flush 1, and 44% more in Flush 2, compared to the compost fill media treatments

**Table 5** Accessibility and cost of materials used in phosphate adsorption

Material	Accessibility and production	Mean cost (£)		Other notes on accessibility	References
		Per 1 t bulk bag	Per mg of PO <sub>4</sub> <sup>3-</sup> removed		
C	Widely available, widespread. UK production <sup>a</sup> 3.5 t/year	75	N/A	Increasing production owing to increasing recycling rates. Agriculture is the biggest compost market	WRAP (2017)
W	Widely available, limited UK production data, often imported	87		Produced as waste in sawn wood production. Reused in sawmill wood processors and for wood fuel	Forestry Commission (2017)
Pp	Not widely available or accessible. Imported from United States	N/A	11.00 <sup>b,c</sup>	Only one supplier in the United Kingdom	
K	Widely accessible. United Kingdom reserves in excess of 60 t, with approximately 1.1 t produced annually		0.23 <sup>b</sup>	Kaolin reserves in the United Kingdom are restricted to Cornwall and Devon. Their exact extent is not available due to commercial reasons. Paper and ceramics are the main markets	British Geological Survey (2009), Cornwall Council (2012), Leclerc and Jackson (2016)
FE	Accessible through imports (~180,000 t/year) from United States, China, Greece, India. Europe produces ~ 21% of global output		0.18 <sup>b</sup>	Exploitable (economic) United Kingdom reserves have been exhausted. Reserves are restricted to the south of England, mainly Bedfordshire, Oxfordshire and the south of Bath	British Geological Survey (2006), Brown <i>et al.</i> (2012), UK Minerals Forum (2014)
FCa–O G–O	Ochre is widely available but not currently accessible. 4500 t/year (natural ochre). Can also be synthesised		0.01 <sup>d</sup>	Ochre is considered waste material as it has no clear end use. It is currently landfilled at cost to the Coal Authority who manage the current UK mining legacy	Sapsford <i>et al.</i> (2015)

Mean cost (£) per mg of PO<sub>4</sub><sup>3-</sup> removed for each treatment = (1/(mean P removed per gram of material in columns)) × mean cost of material

Pp, proprietary product; K, kaolinite; FE, fullers earth; FCa–O, ferrihydrite-ochre; G–O, goethite-ochre.

<sup>a</sup>UK compost production 2014 totalled 3.5 million tonnes, with 3.2 million tonnes certified as PAS 100 (WRAP, 2017).

<sup>b</sup>Does not account for negative PSE results.

<sup>c</sup>Cost does not include air freight costs or import taxes from the United States.

<sup>d</sup>Cost calculated using 2015 dredging and landfill costs incurred by the Coal Authority.

(Supporting Information Table S4). This increased retention time increases contact time between the PO<sub>4</sub><sup>3-</sup> solution and the PSMs, allowing increased PO<sub>4</sub><sup>3-</sup> adsorption.

However, this also potentially increases the desorption of PO<sub>4</sub><sup>3-</sup> from the woodchip fill media. This is demonstrated by the significantly higher leachate PO<sub>4</sub><sup>3-</sup> concentration at Flush 2 (54.0 mg/L) for the un-amended woodchip treatment, following the addition of deionised water, compared to Flush 1 (1.10 mg/L).

### Applicability of the PSMs evaluated for the agricultural sector

This research has highlighted the efficiency of FCa–O and G–O PSMs for PO<sub>4</sub><sup>3-</sup> removal when combined with both compost and woodchip fill media in filter socks. To ensure widespread uptake for diffuse pollution control, both fill media and PSMs need to be cost-effective and easy to access. Table 5 highlights the affordability and accessibility in the United Kingdom of the various materials used within

this study. Furthermore, it frames their cost per mg of PO<sub>4</sub><sup>3-</sup> removed. This can be related to specific water quality targets and the amount of PO<sub>4</sub><sup>3-</sup> removal required to meet these targets.

Both compost and woodchip fill media are widely available. Compost production is increasing annually as household recycling rates increase (WRAP, 2017) with an annual production of 3.2 Mt of PAS 100 QP Compliant compost (WRAP, 2017) and average cost of £75/t. Woodchip is less accessible than compost, as it is generally imported. National production data are limited as woodchip is often produced as waste and reused on site. The mean cost per 1 t bulk bag in 2017 was on average £87, as established from market analysis of suppliers (n = 5).

The PSMs FCa–O, G–O and kaolinite are most widely available within the United Kingdom, with kaolinite produced in the southwest, United Kingdom, with current reserves in excess of 60 Mt (Cornwall Council, 2012). It is widely accessible and affordable to purchase, both direct from suppliers and online. However, the cost per mg of P removed



is more than that for FCa–O, G–O and Fullers Earth. Furthermore, kaolinite was often associated with low and negative PSE.

Natural FCa–O and G–O are widely available within the United Kingdom as 'ochre', found as a precipitate within mining landscapes, mine water treatment schemes and water treatment plants (Hancock, 2004; Sapsford *et al.*, 2015; Shepherd *et al.*, 2016). The cost of ochre per mg of  $\text{PO}_4^{3-}$  removed is as little as £0.01 (Table 5). This is particularly low as currently ochre is not commercially available to buy. The Coal Authority pays to dredge and landfill ochre from their mine water treatment facilities, as it currently does not have an identified 'end-of-waste' certificate. This product certification would need to be undertaken before ochre could be widely accessible and utilised. Synthetic ferrihydrite and goethite is available, but would have financial implications on the cost per mg of P.

The majority of Fuller Earth is imported from central Europe and/or the USA as UK production has ceased owing to the working out of existing planning consents (British Geological Survey, 2006). Nevertheless, Fuller Earth is a relatively low-cost PSM even when import costs are accounted for (£0.18 per mg of  $\text{PO}_4^{3-}$  removed). Fuller Earth may, therefore, be considered a viable low-cost PSM for  $\text{PO}_4^{3-}$  control where  $\text{PO}_4^{3-}$  removal <0.2 mg/L is required. Due to its comparatively low PSE, Fuller Earth may need to be applied at rates >20 kg/m<sup>3</sup> in order to provide consistent and longer term  $\text{PO}_4^{3-}$  removal.

The proprietary product is the least cost-effective of the PSMs, costing £11 per mg of  $\text{PO}_4^{3-}$  removed, even before import costs are considered. Furthermore, as it is currently only widely accessible within the United States, it is not practicable for widespread uptake, especially by the UK agricultural sector where many farmers have low financial margins.

To ensure the commercial viability of the PSM-enhanced filter socks, the cost of utilising the PSMs evaluated needs to be explored further in the context of the site-specific application of PSM-enhanced filter socks.

## Conclusions

(1) The most effective and consistent PSM performance was given by FCa–O and G–O with PSEs of up to 99% across both the compost and woodchip fill media, even with the inherently high  $\text{PO}_4^{3-}$  load released by the woodchip fill media. This is consistent with other studies (Wang *et al.*, 2013; Moreira *et al.*, 2017). FCa–O consistently removed >50% of the  $\text{PO}_4^{3-}$  and was the only PSM which reduced the leachate  $\text{PO}_4^{3-}$  concentration to below the water quality target (<0.05 mg/L).

- (2) FCa–O and G–O were associated with costs of circa £0.01 per mg of  $\text{PO}_4^{3-}$  removed. Consequently, these are recommended for further investigation to optimise their PSE and to be incorporated within filter sock across a range of agricultural runoff scenarios. However, they are currently considered a waste material and end-use certification needs to be obtained.
- (3) Fullers Earth was also a relatively economic (£0.18 per mg of  $\text{PO}_4^{3-}$  removed) and efficient PSM, with a PSE up to 53.4%. Given that its  $\text{PO}_4^{3-}$  removal efficiencies were significantly less than those of FCa–O and G–O, it is recommended that the use of Fullers Earth as a PSM within filter socks is limited to situations where lower removal efficiencies are sufficient to meet water quality targets, or as an interim product whilst FCa–O and G–O end-of-use certifications are obtained. Overall, Fullers Earth has proven to be an important PSM that could be widely utilised in the agricultural sector.
- (4) Kaolinite though widely available, was not an efficient or consistent PSM with widely varying results (removal efficiencies ranging from –30.2 to 23.5%), and a higher cost (£0.23 per mg of  $\text{PO}_4^{3-}$  removed), compared to FCa–O, G–O and Fullers Earth. Similarly, the proprietary product was significantly more expensive (£11.00 per mg of  $\text{PO}_4^{3-}$  removed), and is not widely available in the United Kingdom. Consequently, it is not recommended that kaolinite or the proprietary product be used as PSMs in diffuse pollution control measures in the UK agricultural sector where removal efficiency, cost and reliability are fundamental requirements.
- (5) Combining the PSMs FCa–O and/or G–O PSMs with filter socks provides an opportunity to concomitantly remove sediment, sediment bound-P and orthophosphate from agricultural runoff. This has direct implications for the management of water quality. More research is required to establish the most effective application rate of FCa–O, G–O and Fullers Earth PSMs, for specific  $\text{PO}_4^{3-}$  concentrations, flows and removal efficiency targets.

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