



Robustness and resilience of different solid-liquid separation technologies for tertiary phosphorus removal to low levels by coagulation

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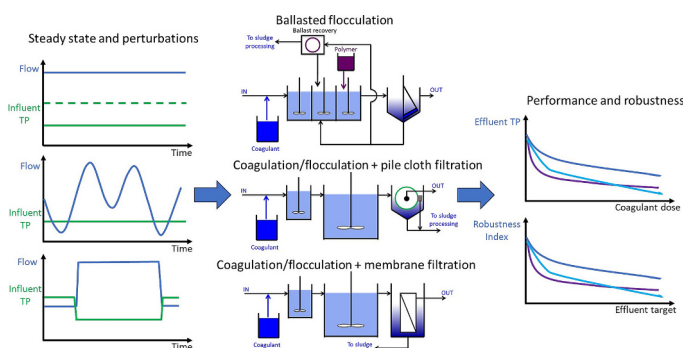
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HIGHLIGHTS

- Steady state and perturbation trials carried out with real wastewaters at demo-scale.
- Ballasted flocculation is very robust and can achieve very low P effluent qualities.
- Pile cloth media filtration is robust for 0.3 to 0.5 mg/L phosphorus targets.
- Membranes cope well with increased phosphorus and solids levels.
- Optimisation of flocculation is critical for robust operation of filtration systems.

GRAPHICAL ABSTRACT



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ABSTRACT

In this study, three tertiary solid separation technologies were assessed on their robustness and resilience against an effluent phosphorus target of <0.3 mg P/L at steady state and dynamic conditions. The ballasted flocculation system was found to be very robust at delivering the low P target. Alternatively, cloth filtration provided a more sustainable option for less strict consents of sub 0.5 mg P/L. The effluent from the membrane system was more variable but it was shown to meet the low consents even with increased phosphorus and solids content in the feed. A molar ratio of 1.37 Fe: P was shown to be sufficient to meet the P target at short contact times as with the ballasted flocculation process. It was highlighted that optimisation of up-stream flocculation can be a considerable factor for consistent performance. Overall, the study determined key attributes of the different technologies tested providing valuable insights for technology selection at full scale.

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1. Introduction

Phosphorus (P) discharge standards from sewage works relate to the need to manage the potential ecological impacts of effluent discharges. The specific levels vary considerably across the world and are often directly linked to the characteristics of the receiving waterbody. In Europe, recent revision of the required standards is seeing a reduction in the annual average phosphorus concentration from levels of 1.0–2.0 mg P/L to between 0.1 and 1.0 mg P/L with many sites expected to meet levels around 0.3 mg P/L (EU Water Framework Directive, 2000). Traditional practice for targets of 1 mg P/L and above is to design and operate treatment works to meet 50 % of the target level to ensure compliance (Vale, 2024). However, below 1 mg P/L, targets of 70–80 % of the permitted value are considered operationally appropriate such that for a 0.3 mg P/L, the site would need to deliver an effluent with an annual average of between 0.21 and 0.24 mg P/L. Stricter discharge consents are common place in the USA where levels can be significantly below 0.1 mg P/L (Lee et al., 2015; Ragsdale, 2007).

A large proportion of the wastewater treatment sites utilise chemical precipitation with metal salts (Fe or Al) for all, or part, of the required removal. The mechanisms of removal comprise a combination of precipitation of metal-phosphate-hydroxo complexes, adsorption onto precipitated surfaces and aggregation of colloids (Bratby, 2016). Phosphorus exists in many forms including ortho-phosphate (ortho-P), condensed forms (pyrophosphates, triphosphates) and poly-phosphates with the relative distribution impacted by concentration, pH and ligand reactions with the other components in the wastewater. Further, transformation occurs during the aerobic secondary processes such that the majority of phosphorus is in the ortho-phosphate form once it reaches the tertiary stage (Bratby, 2016). The removal efficacy is highest for ortho-P form. Removal of the other phosphorus fractions is associated with a significantly higher coagulant demand. Therefore, it is common practice to dose coagulant during or after the biological process rather than in the primary stage so that dose requirements can be minimised. By dosing at a later stage in the treatment train, in a cleaner effluent, chemical demand will also be decreased due to the reduction in competition between P and organic matter. The dose expressed as a stoichiometric mole ratios of Metal: P is reported to vary significantly between 0.5 and 77 (Table S1 in supporting information). The relative importance of direct precipitation decreases as the target residual levels becomes lower with removal to below 1 mg P/L thought to be predominantly associated to adsorption and colloidal aggregation. Accordingly, the relative dose requirements increase significantly as the target level is reduced. To illustrate in the case of Alum dosing, 83.5 mg/L were required to reduce phosphorus levels from 5.17 to 0.97 mg/L at the head of a contact stabilisation plant. In comparison, 125 mg/L were required to reduce the total phosphorus (TP) levels from 2 to 0.05 mgTP/L in an oxidation pond effluent (Bratby, 2016).

Consequently, whilst compliance is possible through single point dosing into the crude wastewater and/or the main biological process, the coagulant doses to reach sub 1 mg P/L are substantial (Bratby, 2016; Murthy et al., 2005). Accordingly, it is becoming more common to meet sub 1.0 mg TP/L standards by incorporating a tertiary coagulation step linked to a range of different solid liquid separation systems (Table S1 in Supplementary Information). These include traditional tertiary treatment technologies used for solids removal such as depth filters and cloth filters as well as advanced filtration with membranes and, a relatively recent technology, ballasted flocculation where a dense material, the ballast, is incorporated in the flocs with the addition of flocculant (e.g. polymer) to increase the density of the particles for faster settling. The different options operate with different removal mechanisms for the precipitated solids and are effective over different particle size ranges. For instance, membranes sieve out particles larger than the opening, typically 0.05 μm (Bill et al., 2011). In contrast, depth filters work by a combination of collision and attachment mechanisms with minimum removal associated with particle sizes around the 0.45 μm level (Hahn

and O'Melia, 2004). Pile cloth media filters sit in between the two with entrapment within the pile and through the backing material and the formation of a dynamic cake layer aiding removal during the filter cycle (Grabbe et al., 1998). In contrast, ballasted systems operate through sedimentation and hence small particles are poorly removed in line with Stokes' law.

The fact that they operate under different mechanisms suggests that the different separation processes will be impacted differently by variations in the feed water flow and composition during steady state operation and respond differently to external or internal perturbations. Consideration of such issues connects to concepts of robustness and resilience as commonly used in ecological systems (Janssen et al., 2007). Whilst different definitions are used, the most appropriate to the current discussion are that robustness relates to the consistency of the output against a variable input and resilience relates to the ability of a technology to recover from a perturbation that has caused a deterioration in the output (Nazif and Karamouz, 2009). There is a paucity of translation to treatment technology assessment, especially in relation to predicting future capability. However, examples exist for depth filters in drinking water treatment (Hartshorn et al., 2014; Li and Huck, 2008), clarifiers (Huck and Coffey, 2004) and aerated constructed wetlands for wastewater treatment (Butterworth et al., 2013). Assessment of robustness is derived by examining the cumulative distribution of the effluent concentration whilst operated under steady state conditions. This can be converted in a quantifiable index through a number of performance indices (Hartshorn et al., 2014). The most common is the robustness index although it is known to being heavily dependent on the goal target and the percentile levels chosen (Huck and Coffey, 2004). However, when comparing different options, these weaknesses are less important as the comparison is set to the same parameters and as such focus can be given to the relative difference and the overall profiles.

Previous work has mostly been associated with achieving ultra-low P effluents of between 0.017 and 0.15 mg TP/L from influent levels that varied between 0.16 and 4 mg TP/L (Table S1 in Supplementary Information). There is also a paucity of direct comparison between technology options limiting decision-making based on performance efficacy. Where these do exist (DeBarbadillo et al., 2010; Lee et al., 2015; Fundneider et al., 2020) the trials are again focused on delivering ultra-low discharge standards (sub 0.1 mg P/L), using high coagulant doses making transition to the current challenges to meet sub 1 mg P/L in Europe very difficult.

There is a clear gap in understanding the impact of process configuration combining coagulation with different solids separation technologies on the efficacy and robustness to achieve sub 1 mg TP/L for discharge. For this, the current study compares the performance of three demonstration scale, coagulant based tertiary treatment options (i.e. coagulant with separation by membrane, pile cloth media filter, and ballasted flocculation) during steady state and perturbations and benchmarks their performance against the more traditional coagulation/sand filter approach.

2. Materials and methods

2.1. Materials

Three coagulant-based technologies: ballasted flocculation, pile cloth media filtration, and membrane filtration (Figure S1 in Supplementary Information) were installed as tertiary treatment at a wastewater treatment plant in Leicestershire, UK. The systems were studied as part of a broader trial, with other technologies, on the removal of P to low levels and ultimately informed an overall strategy. All the technologies were operated as per the supplier's instructions. The site has no primary treatment and iron-dosed oxidation ditches (OD) as secondary treatment. For the purpose of the trial, influent ortho-P to the pilot systems was supplemented to 1 or 2 mg P/L with KH_2PO_4 as required (Table S2 in Supporting Information). A second set of tests were

performed using tankered secondary treated wastewater from the trickling filter-based site that contained the benchmark depth filter technology.

The ballasted flocculation technology (BF) (CoMag®, Evoqua Water Technologies, UK) consisted of three mixing tanks and a clarifier (each 270 L) (Figure S1a in Supplementary Information). Over the range of flow rates tested, the surface overflow rate ranged between 5 and 15 m³/h/m² of tank corresponding to hydraulic residence times between 5.5 and 15 min for the whole system. The trials were conducted for two coagulants: ferric chloride (FeCl₃) and ferric sulphate (Fe₂(SO₄)₃; Fe—S) (Table 1). The coagulant was dosed into an in-line static mixer. The ballast, magnetite, reported to have a composition of 86–99 % Fe₃O₄, a density of 4.6–5.1 kg/L and an average particle size d₅₀ of 64 ± 4 µm when dispersed in the wastewater and a polymer (anionic polyacrylamide) were dosed at 4 g/L and 0.5–1 mg/L, respectively. Each of the chemical compounds was added subsequently to each of the mixing tanks. In the clarifier, 25 % of settled sludge was recycled back into the system and the rest underwent shear mixing to recycle the ballast through a magnetic drum back into the treatment train.

The pile cloth media filtration technology (CF) had a fast-mixing coagulation (330 L) and a flocculation tank (1650 L) upfront. The mixing speeds were optimised throughout the trial. The mixing rate of the fast-mixing coagulation tank was 200 RPM for the first 185 days and was then increased to 290 RPM for the remaining 22 days of the trial. In the flocculation tank, the mixing rate was 130 RPM during the first 125 days of operation which was then reduced to 35 RPM for 4 days and after that to 25 RPM for the remaining 78 days of the trial. Over the range of

flow rates tested, the hydraulic residence times varied between 1.4 and 5.5 and 6.9–27.5 min in the fast mixing and flocculation tanks, respectively. A proprietary microfibre pile cloth with a surface area of 2 m² was fixed in a drum filter (TF2, Mecana®, Hydrok, UK) (Figure S1b in Supplementary Information). Under operation, solids accumulate on the cloth restricting flow through, leading to a rise in water level and ultimately triggering backwash during which the drum rotates a full turn and deposited solids are removed with a suction pump from the surface of the cloth. FeCl₃ was the coagulant used for this technology (Table 1). For the flow rates tested, the hydraulic and solids loading rates ranged between 1.8 and 7.2 m³/h/m² of cloth and 0.013–0.052 kg TSS/h/m² of cloth, respectively.

The membrane filtration (MF) technology consisted of an ultrafiltration hollow fibre membrane cartridge (Puron MP®, Koch Membrane Systems, Germany) (pore size = 0.03 µm, Polyester reinforced PVDF) with the same fast mixing (330 L, 290 RPM) and slow mixing (1650 L, 25 RPM) tanks upstream as with the cloth filter (Figure S1c in Supplementary Information). The mixing rates were not varied during this trial. The membrane had a surface area of 51 m² and was configured in an outside-in flow and operated in dead end mode at fluxes between 15 and 50 L/h/m² of membrane. Every 20–30 min of filtration, backwashes were induced for 170 s. Every 2 days, acid chemical cleans (pH 2–2.5) were carried out to reduce transmembrane pressure below 600 mbar, remove remaining deposits from the membrane surface and to maintain permeability. The membrane filtration was operated with two coagulants: FeCl₃ and dialuminium chloride pentahydroxide (Al₂Cl(OH)₅, PAC) (Table 1).

The technologies were benchmarked against a full-scale depth filter which was adapted to include upstream coagulant dosing located at a site comprising primary settling tanks, trickling filters (TF) as the main biological process and humus tanks from which the sand filter were fed. The filter was a continuous backwash upflow moving bed filter containing 1 mm sand operated at a surface loading rate of 8.96 m³/h/m² of tank (Blue Pro®, Blue Water, USA) (Figure S1d in Supplementary Information). At the TF site, the sand filter (SF) consisted of 5 filters (each 25 m³, 3 m diameter) which were operated at an average flow rate of 17.6 L/s per filter. The flow rate was adapted to the flow on the main site. The coagulant, Fe₂(SO₄)₃, was dosed flow proportionally in an inline static mixer directly upstream of the filters, relying on pipe flocculation, and ranged between 10 and 14 mg Fe/L (Table 1).

2.2. Methods

The trials were carried out at steady state (fixed flow and influent P concentration), treating wastewater from both sites, and with perturbations with varying coagulant doses, influent P concentrations and flow patterns (storm, diurnal flow pattern) (Table 1). Results were evaluated against an effluent P target of 0.3 mg/L unless otherwise noted. Influent and effluent samples were taken every 15 min and analysed for ortho-P and ferric concentrations as well as turbidity (online analysers, ABB, UK). Additionally, grab samples were taken periodically and further analysed for TP, COD and NH₄-N using test kits (Hach Lange, UK). Fractions for TP were further analysed by dividing the samples into unfiltered and filtered through 1.2 µm and 10 kDa representing solid, colloidal and dissolved fractions, respectively. Suspended solids (TSS) were determined by filtering 250 mL of a sample through a 1.2 µm glass fibre filter and drying in an oven at 105 °C.

2.2.1. Steady state

CF, BF and MF were run at unchanged operational conditions for at least two weeks to evaluate their robustness. The robustness index (RI) was calculated according to Hartshorn et al. (2014) for a range of P targets between 0.05 and 0.9 mg P/L using Eq. (1).

$$RI = \left[\left(1 - \frac{G\%}{100} \right) \cdot \frac{P_{90}}{P_{50}} \right] + \left[\frac{P_{50}}{P_{Goal}} \cdot \frac{G\%}{100} \right] \quad (1)$$

Table 1

Set parameters for tests done with coagulation technologies. The used coagulant was FeCl₃ for pile cloth media filtration (CF), ballasted flocculation (BF) and membrane filtration (MF) unless otherwise noted. The sand filter (SF) was operated with ferric sulphate (Fe—S).

Operational test	Technology	Influent ortho-P [mg/L]	Coagulant dose [mg/L]	Polymer dose [mg/L]	Flow rate [L/s]
Steady state	CF (19 days)	1	5	NA	2.5
	BF (17 days)		8	1	1.5
	MF (37 days)		5	NA	0.57
	SF (15 days)	0.93 ^a	10–14	NA	17.6 ^b
Coagulant dose response	CF (49 days)	1	1–9	NA	2.5
	BF (45 days)		3–8	1	1.5
	MF (77 days)		1–6	NA	0.57
Storm	CF (3 days)	0.5–2	12–6	NA	1–4
	BF (3 days)		8	1	1.5–3
Diurnal flow profile	CF (10 days)	1	4	NA	1.3–3.3
	BF (8 days)		5 (Fe—S)	1	1.2–2.9
High influent P	CF (6 days)	2	8–10	NA	2.5
	BF (6 days)		8	0.5	2
	MF (5 days)		6–7 (PAC)	NA	0.57
	CF (19 days)		9	NA	1
TF site feed	BF (17 days)	1.25 ^c	8	1	1.5
	MF (37 days)		6 (PAC)	NA	0.57
	SF (15 days)	0.93	10–14	NA	17.6

^a The influent P concentration was not altered on this site and therefore the measured value of orthophosphate is given here.

^b This was the average flow of each sandfilter cell. There were 5 cells in total with variable on/off depending on flow pattern on the main site.

^c Based on average measured ortho-P concentration.

where G% represents the percentile at target effluent P concentration, P_{Goal} , P_{90} and P_{50} are P concentrations at the 90th and 50th percentile, respectively. A lower RI value represents a more robust system towards the set goal. Performance of the CF, BF and MF were also compared to the previously described SF. Within this context, RIs of below 1 are considered good and below 0.5, very good.

2.2.2. Perturbations

2.2.2.1. Coagulant dose response. Coagulant dose response curves were obtained from operating the systems at varied coagulant doses while maintaining a constant influent P concentration (1 mg P/L) and flow rate. The data from these tests were analysed statistically (Statistica 13) using a Box-Cox transformation for effluent P values. The transformed results were then plotted with an ANOVA factorial general linear model where coagulant doses and technology type were used as categorical variables. A post-hoc Fisher LSD analysis was used to determine significant differences ($p < 0.05$) between the three technologies.

2.2.2.2. Change in influent characteristics. An increase in influent P concentration from 1 to 2 mgP/L was tested for 4–6 days at fixed flow rates and the CF, BF and MF were fed with the wastewater from the TF site to trial performance and for comparison to the SF (Table 1).

2.2.2.3. Dynamic operation. Storm tests were run for 4 h and 24 h with a rapid increase from a low flow rate (CF: 1 L/s and BF: 1.5 L/s) to a high flow rate (CF: 4 L/s, BF: 3 L/s) while simultaneously diluting influent P from 2 mg/L to 0.5 mg/L (Figure S2 in Supplementary Information). Also, diurnal flow profile tests were run for 4 days to simulate a real-life wastewater flow with higher flows in the mornings and evenings and lower flows during the day. Flows varied between 1.3 and 3.3 L/s and 1.2 and 2.9 L/s for CF and BF, respectively. The MF was not operated under storm or diurnal flows as it would typically be designed based on peak flow operation.

3. Results and discussion

3.1. Steady-state operation and robustness

All four technologies demonstrated the ability to meet a 0.3 mg P/L discharge level, target for a new consent to be implemented at the site, during the steady state trials (Fig. 1). To illustrate, the 50th Percentile, used here as a proxy for the annual average, was 0.02, 0.03 (below

detection limit of 0.05 mg/L), 0.14 and 0.18 mg P/L (as ortho-phosphate) for the SF, BF, MF and CF, respectively. Fractionation tests revealed that 81 % of the incoming P was in the dissolved form, 1 % was colloidal P and 18 % P was associated to solid matter. Conversion of the data to an operational TP standard is most conservatively assessed by subtracting the difference between TP and ortho-P in the influent, adding this value onto the effluent ortho-P values (assuming no non ortho-P removal) and then reducing to 70 % of that value to provide an operational buffer. This would set the ortho-P effluent target to 0.21 mg P/L. In this case, they still all met the standard at the 50th percentile, with the SF and BF well below the target but the CF was very close to the value at 0.18 mg P/L. Effluent total phosphorus spot sample measurements during the steady state trials reported median levels of 0.05 ± 0.05 , 0.083 (based on 7 samples taken during steady state operation that varied between 0.07 and 0.28 mg TP/L), 0.17 ± 0.13 and 0.30 ± 0.27 mg P/L for the BF, MF, SF and CF, respectively, in which case the CF would not meet the consent. Consideration of the robustness curves indicates robust profiles for the SF and BF with a larger proportion of the data associated to a steep vertical curve, showing that the vast majority of the samples remained at very low and stable concentrations, followed by a tail at very high percentile values (Fig. 1). For instance, the 90th percentile values were 0.03 and 0.07 mg P/L for the SF and BF, respectively, with maximum values of 0.54 and 0.84 mg P/L. For the CF, a similar trend was observed with a steep curve and tail at higher percentiles but all for higher concentrations. Indeed, for up to the 90th percentile, the concentration ranged between 0.1 and 0.24 mg P/L and increased to a maximum of 0.56 mg P/L beyond this. In contrast, the MF profile was indicative of a less robust system where the curve was less vertically inclined and spread over a broader range of effluent ortho-P values. To illustrate, effluent concentrations ranged between 0.01 and 0.27 mg P/L up to the 90th percentile with a maximum value at 0.34 mg P/L.

The corresponding robustness index values (Fig. 2), assuming a target of 0.21 mg P/L as ortho-P at the 50th percentile, were 0.18, 0.24, 0.95 and 0.97 for the SF, BF, MF and CF, respectively. This demonstrates a greater robustness for the SF and BF and validates the results from the cumulative curves described above (Fig. 1). Indeed, big differences in robustness can be seen between the technologies, especially at lower targets. This clearly establishes that the SF and BF technologies are able to consistently meet low effluent targets. The trends for the CF and MF are similar with high RI values for the low effluent targets. However, the MF displayed the highest of RIs of 2.0 and 1.8 for the lowest targets values of 0.05 and 0.1 mg P/L, respectively. This again supports the findings from the cumulative curves (Fig. 1) as the MF displayed more

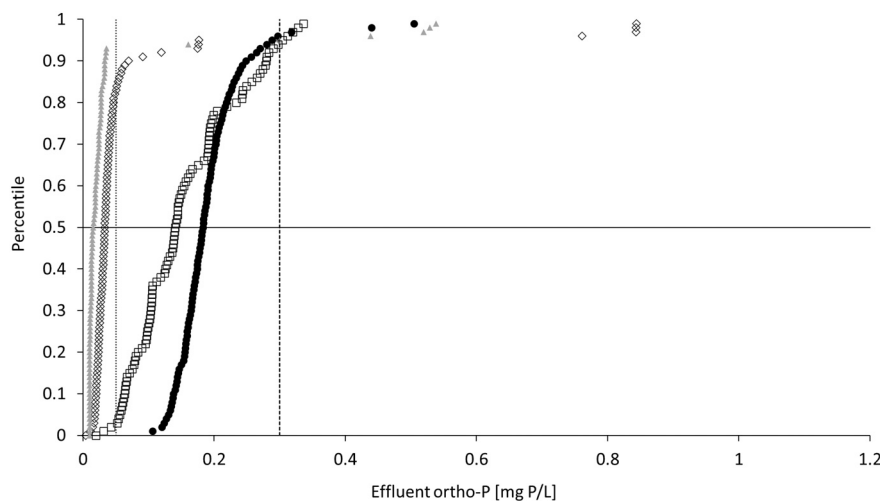


Fig. 1. Cumulative effluent qualities at steady state for the sand filter (▲), cloth filter (●), ballasted flocculation (◇) and membrane filtration (□) with P detection limit (.....) and TP target (—).

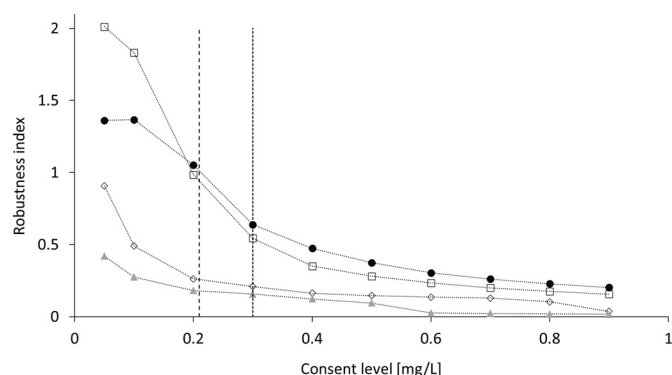


Fig. 2. Robustness indices against potential P consents for the sand filter (▲), cloth filter (●), ballasted flocculation (◇) and membrane filtration (□) with the TP (—) and ortho-phosphate (---) targets for the site of interest.

variable effluent qualities while the CF was shown to deliver more consistent effluent quality around 0.3 mg P/L. The RI values for all technologies then converge at higher target values (Fig. 2) demonstrating that all technologies are robust at delivering effluent qualities above 0.4 mg P/L.

It has to be noted though that the SF was operated at higher coagulant doses (10–14 mg Fe/L, 15.9–22.3 Fe:P molar ratio) than the other technologies (5–8 mg Fe/L, 2.29–3.67 Fe:P molar ratio). In addition, the flocculation setup used in the BF appears better optimised than that used for the MF and CF such that sub optimal flocculation and excessive floc breakage are likely to have contributed to the poorer performance of the two filtration processes. To illustrate, when the mixing speed in the upstream flocculation tank to the CF was reduced at an earlier stage of the trials from 135 rpm to 35 rpm and then 25 rpm, average P removal changed from 70.7 % to 80.6 % and then 71 %, respectively. This indicates the importance of the mixer speed on final P concentration, as over or under mixing impacted the performance due to possibly too high shear breaking the flocs formed or low shear limiting the opportunity for collision and attachment to form larger flocs. Previous trials exploring the different components within the BF have revealed that the combination of coagulant, polymer and ballast are able to remove more phosphorus than coagulant alone (Murujew et al., 2020). This is attributed to the polymer sweeping up colloids that were originally in the water and those generated by the chemical precipitation. The combination results in large particle sizes with small number of fines compared to coagulant alone and thus is well suited to achieve very low residual phosphorus concentrations. This is supported by the respective residual solids levels observed during the trials which were 8.47 ± 5.09 , 4.13 ± 1.35 , 1.03 ± 0.78 and 9.00 ± 1.11 mg/L for the SF, BF, MF and CF respectively. However, the SF was also able to meet a very low

effluent concentration and achieved this with coagulant alone, albeit at a much higher dose. Either way this suggests that effective flocculation is critical in ensuring robust treatment and the use of low polymer dosing is likely to enhance P removal for all separation technologies.

3.2. Varied influent characteristics

The wastewater at the test site was low in suspended solids and this has been shown to inhibit effective flocculation (Havlicek, 2018), likely to have impacted the observed results from the steady state trials especially in relation to the CF and MF systems as the BF adds additional particles in the form of a ballast and recycled sludge (Murujew et al., 2020). Accordingly, wastewater was tankered from the TF site where the SF was located and then run through the other technologies (Table 2). Although the influent was from the same site, a difference in ferric, TSS, ammonia-N and COD concentration was observed between the influent analysed directly at that site and when it was analysed after transport and so the results are presented relative to the specific influent that was treated during the trial (Table 2).

Ortho-P removal, based on median concentrations, was in order of performance 98.2 %, 92 %, 84.7 % and 83.9 % for SF, MF, BF and CF, respectively, showing a better performance for the membrane than the ballasted flocculation process which is likely due to the change to an Al based coagulant in the MF (Langer and Scherman, 2013) which was also observed during trials with the oxidation ditch effluent as feed. At equivalent conditions, values of 94.5 %, 88.9 % and 81.8 % for MF, BF and CF, respectively, were observed during the trials with the oxidation ditch effluent indicating that the more polluted wastewater from the trickling filter site resulted in a slight deterioration in treatment performance for the BF while leading to a slight improvement for the MF and CF. The associated median value for TP were then 0.15, 0.17, 0.27 and 0.33 mg P/L for MF, SF, CF and BF, respectively, indicating that the BF and potentially the CF were not able to meet the operational target of 0.3 mg P/L, although this is for a low sample number. Interestingly, the higher levels of solids present in the TF effluent are likely to have helped generate larger flocs in the coagulation/flocculation step before the MF and CF, making separation by the filtration technologies more efficient. Alternatively, the change in water characteristics from the OD effluent to the TF effluent has negatively impacted the performance of the BF. With again the formation of larger but more fragile flocs in the TF effluent, the high shear (high mixing to maintain the ballast in suspension) and short contact time resulted in floc breakage and carry over in the treated effluent.

Previously reported data on trials of individual processes indicates better performance than observed in the current trial. For instance, a ballasted flocculation trial with an influent of 1 mg TP/L was reduced to 0.04 mg TP/L with a coagulant dose of 20 mg Al/L of poly aluminium chloride (PACl) and 0.8 mg/L of polymer (Lee et al., 2015). Similarly, in

Table 2

Effluent wastewater characteristics with the trickling filter (TF) effluent as influent. Data given as median values with total number of samples.

	Ortho-P [mg/L]	TP [mg/L]	Fe [mg/L]	TSS [mg/L]	NH ₄ -N [mg/L]	COD [mg/L]
Influent SF (n = 30)	0.84 ^a	1.50	0.28 ^b	13.75	3.24	45.0
SF (n = 30)	0.02	0.17	0.33	6.99	2.85	19.5
Influent CF/ BF/ MF	1.30 (13)	1.72 (13)	2.51 (8)	27.30 (12)	4.67 (8)	52.7 (13)
CF	0.21 (23)	0.27 (2)	1.50 (23)	10.35 (24)	5.82 (2)	28 (2)
BF	0.20 (19)	0.33 (3)	1.67 (20)	7.50 (19)	4.30 (3)	26.2 (3)
MF	0.10 (8)	0.15 (8)	0.14 (42)	1.9 (8)	–	19.8 (8)
Removal rates [%]						
SF	98.2	88.4	–	49.2	12.1	56.7
CF	83.9	84.0	40.2	62.1	–	46.9
BF	84.7	81.1	33.7	72.5	7.8	50.3
MF	92.0	91.3	94.3	93	–	62.4

^a In this case ortho-phosphate was measured where the sample is filtered through a 0.45 µm filter and then analysed using a colorimetric method.

^b Ferric was analysed with ICP instead of a colorimetric method.

a pilot scale depth filter trial using a 5.7 mg Al/L dose of polyaluminium silicate sulfate a residual of 0.06 mg TP/L was achieved from an initial concentration of 1.5 mg TP/L (Bratby, 2016). However, the required conditions vary considerably due to the phosphorus species present and the nature of the coagulant. For instance, another depth filter trial treating a 0.135 mg TP/L influent required up to 7 mg Fe/L (28.7 Fe:P molar ratio) of ferric chloride to reduce the phosphorus concentration down to 0.017 mg TP/L. When elevated concentrations of coagulant are used, the resultant metal speciation changes with increasing production of polymeric species and a reduction in the efficacy of removal such that higher Fe:P dose ratios are required (El Samrani et al., 2004).

3.3. Perturbation: coagulant dose response

Dose response trials with the three pilot technologies revealed a decrease in residual ortho-P at low doses which thereafter stabilised (Fig. 3). To illustrate, in the case of the CF, the ortho-P concentration decreased from 0.46 mg P/L to 0.26 mg P/L between doses of 1 and 3 mg Fe/L. Whereas, between doses of 4 to 8 mg Fe/L, the residual concentration stabilised between 0.17 and 0.22 mg P/L (Fig. 3). A similar response was observed in the case of the MF where the effluent concentration dropped from 0.51 mg P/L to 0.2 mg P/L between coagulant doses 1 and 2 mg Fe/L. At higher doses of 3 to 8 mg Fe/L the residual concentration fluctuated between 0.2 and 0.04 mg P/L. In the case of BF, only doses of 3 mg Fe/L and above were tested and, at these doses, a fluctuating residual concentration of between 0.12 and 0.08 mg P/L was observed. Previous jar test trials with the different BF components revealed that performance was stable down to coagulant doses of 5 mg Fe/L, consistent with the current findings (Murujew et al., 2020). Overall, to meet an ortho-P target of 0.21 mg P/L, a coagulant dose of 3 to 4 mg Fe/L appears appropriate for the BF and MF and a minimum dose of 5 mg Fe/L seems necessary in the case of CF. This equates to dose molar ratios (Me:P) of between 1.37 and 1.83 and of 2.29 respectively which is towards the lower end of commonly reported values for tertiary treatment but consistent with standard practice in general phosphorus precipitation (Table S1 in Supporting Information). At these doses the ferric species are predominately iron dimers linked to a single phosphate anion (El Samrani et al., 2004). To achieve sub 0.1 mg P/L, residual substantially higher doses are required as the incorporation of phosphate into the precipitate becomes less effective and the coagulant starts to form progressively more poly-hydrolysed species. Interestingly, statistical analysis showed that there are significant differences in the data set of varied coagulant doses for BF, MF and CF indicating that the different solid-liquid separation mechanisms involved result in different treatment efficacies. Detailed understanding of this linkage is currently limited and represents an area of important future activity to fully

optimise such systems by properly tailoring the coagulation-flocculation process to the downstream mechanism of separation.

3.4. Perturbation: dynamic operation and stress testing

The systems were then tested against dynamic response to a change in phosphorus concentration, dose or flow (Figs. 4, 5, 6). A step change in the influent phosphorus concentration from 1 to 2 mg P/L resulted in a sharp increase in the effluent phosphorus concentration with the CF but was not observed for the MF or the BF systems. In the case of the CF, the effluent concentration increased to 0.49–1.15 mg P/L which compares to the slight increase seen for the other technologies with peaks of 0.07 and 0.17 mg P/L as ortho-P (Fig. 4a). In response, the coagulant dose to the CF was increased from an initial value of 4 to 8 mg Fe/L. The residual phosphorus initially declined to a low of 0.57 mg P/L before increasing again. The coagulant dose was then increased to 9 and 10 mg Fe/L on days 2.5 and 5.5, respectively. This did not result in a sustained reduction in the effluent P concentration as it was found to fluctuate between 0.63 and 1.5 mg P/L. Interestingly, the fluctuations were found to follow a daily pattern with peaks during the day and trough during the night. The better performance observed at night is most probably due to the less complex nature of the wastewater reaching the sewage works over that period as opposed to during the day (Raboni et al., 2013) with increased levels of cleaning products and detergents which would compete with P for the coagulants. In the case of the MF, it is noteworthy that when the coagulant was changed to PACl an improved performance was observed compared to tests with FeCl₃ during the dose response test (Figs. 4b). Although analysis of the impact of coagulant type on performance was not an objective of this work, this highlights an aspect worth examining in the future.

The BF and CF were then exposed to dynamic flow events in the form of a 4- and 24-h storm whereby the flow increased by 300 and 400 % for the BF and CF, respectively, and the phosphorus concentration decreased by 75 % (Figure S2 and 5). Before the storm simulation started, effluent P from the CF ranged between 0.35 and 0.5 mg P/L, thus already above the target of 0.3 mg P/L (Fig. 5a). At 5.75 h, half an hour after the maximum flow rate was reached, the effluent P dropped down to 0.135 mg P/L and decreased further to 0.075 mg P/L at 7.25 h. At the end of the storm simulation, i.e. 8.5 h, the effluent P was 0.07 mg P/L and started increasing up to 0.3 mg P/L at 11 h. Because the highest P values have been reached outside of the storm flow conditions, i.e. before 4.25 h and after 8.5 h, but during the high influent P setting of 2 mg/L, it appears that the P concentration plays a more significant role in CF performance than the flow rate. It has to be noted that the coagulant dose was adjusted between 6 and 12 mg Fe/L when the P was at minimum or maximum, respectively (Table 1). This equates to molar ratios

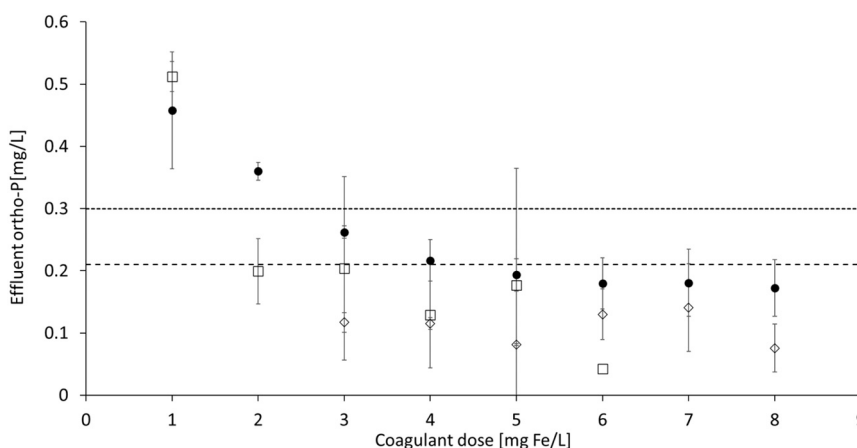


Fig. 3. Coagulant dose response for the pile cloth media filter (●), ballasted flocculation (◇) and membrane (□) against the TP (—) and ortho-phosphate (---). Average values with error bars ± 1 standard deviation.

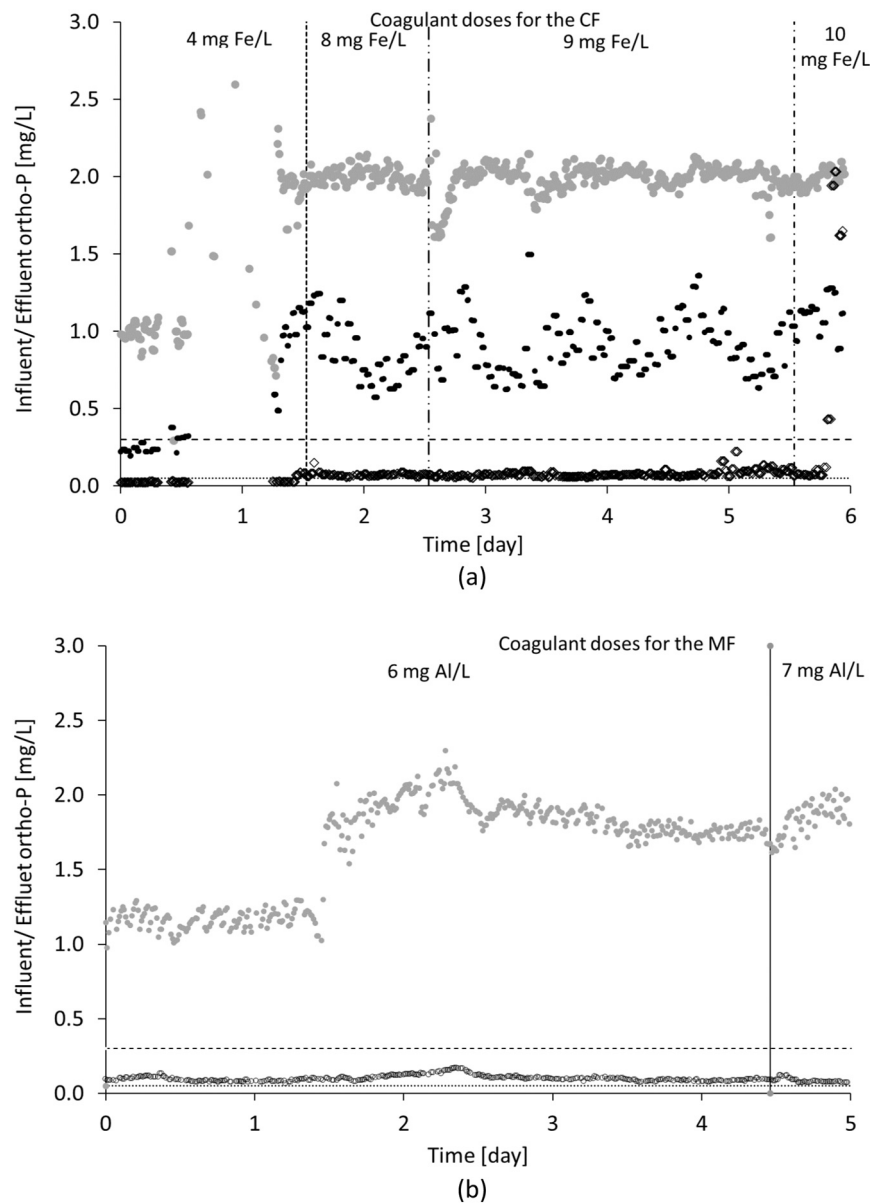


Fig. 4. Impact of increased influent P concentration from 1 to 2 mg P/L on treatment performance with the influent P (●) and the pile cloth media filtration (CF) (●) and ballasted flocculation (BF) (◇) effluents in (a) and the membrane (MF) (□) effluent for in (b). For CF, the coagulant dose was 4 mg Fe/L first and then increased to 8 mg Fe/L (—), 9 mg Fe/L (---) and 10 mg Fe/L (....); BF was dosed with 8 mg Fe/L and 0.5 mg/L polymer; MF had a coagulant dose of 6 mg Al/L later increased to 7 mg Al/L (—). P detection limit (.....) and TP target (---).

between 3.32 and 6.64 Fe:P for high and low influent P, respectively, suggesting that under-dosing occurred at 2 mg/L influent P conditions. Nevertheless, the performance of the CF system remained relatively stable during the storm simulation showing that the technology can be resilient in dynamic situations. The response to the prolonged storm (24 h) was similar to the 4-h storm simulation, with effluent P ranging above the P target before and after the 24 h storm at values between 0.33 and 1.11 mg P/L and 0.33–1.01 mg P/L, respectively (Fig. 5b). At 12.75 h, i. e. 0.15 h before reaching maximum flow, the CF reached a peak of 0.78 mg P/L in the effluent which quickly decreased down to 0.13 mg P/L at 13.75 h. The flow rate decreased between 32.75 h and 34.75 h, the end of the storm simulation, and during that time the effluent P ranged between 0.12 and 0.33 mg P/L.

For the BF, the effluent P ranged between 0.04 and 0.14 mg P/L during the 4-h storm simulation which suggests that this system is robust to sudden flow changes (Fig. 5c). During the 24-h storm, the BF showed an increase in effluent concentration to 0.3 mg P/L at 10.75 h with a

subsequent decrease to 0.08 mg P/L at 14.75 h (Fig. 5d). Performance was kept stable until the end of the storm at 34.75 h but was followed by an increase to 0.35 mg P/L at 36.25 h which does not seem to be a dosing issue (2.21 Fe:P molar ratio) because the “High influent P” test was conducted at the same coagulant dose (8 mg Fe/L) and did not show adverse impact on performance (Fig. 4a). Overall, storm conditions did not appear to cause performance disruptions in the BF suggesting that the flocculation system is adequate for the changes presented. This aligns to the reported fast reaction time of the hydrolysing ferric coagulant which forms precipitates within seconds (Bratby, 2016). The same was observed for the CF system where the change in influent concentration appeared to have more impact than flow dynamics. Indeed, an increase in flow led to a reduction in the contact time in the systems but, as stated above, the speed of reaction is very fast which meant that in both systems good removals could still be achieved, especially with low incoming P concentrations. Ultimately this reflects the need to optimise both the dose and the mixing conditions for effective flocculation.

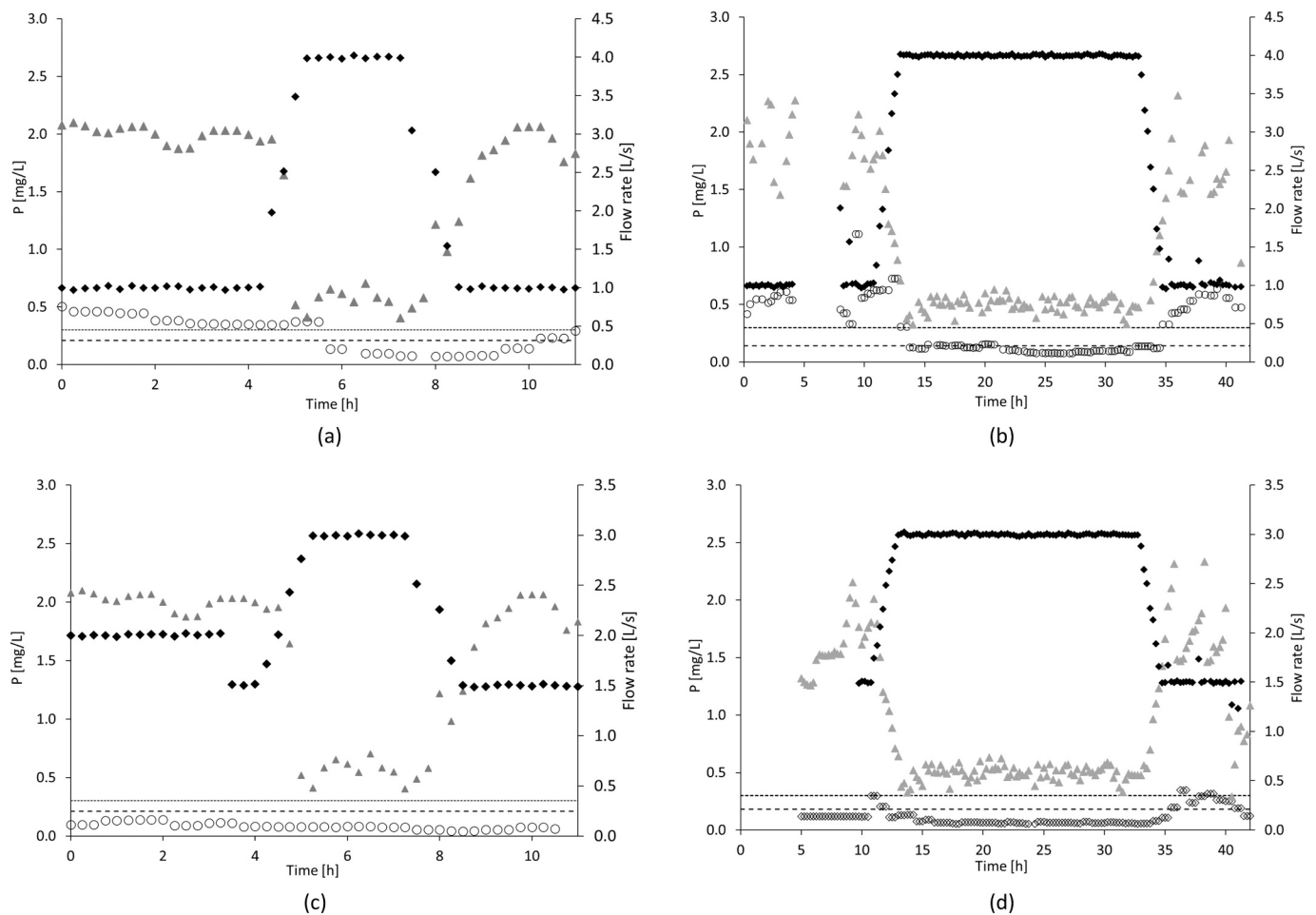


Fig. 5. Impact of storm simulations on performance of the pile cloth media filtration (CF) and ballasted (BF). Influent P (▲), effluent P (○) and flow rates (◆) during (a) 4 h Storm test for CF (b) 24 h Storm test for CF (c) 4 h Storm test for BF (d) 24 h Storm test for BF with TP (.....) and ortho-phosphate (----) targets.

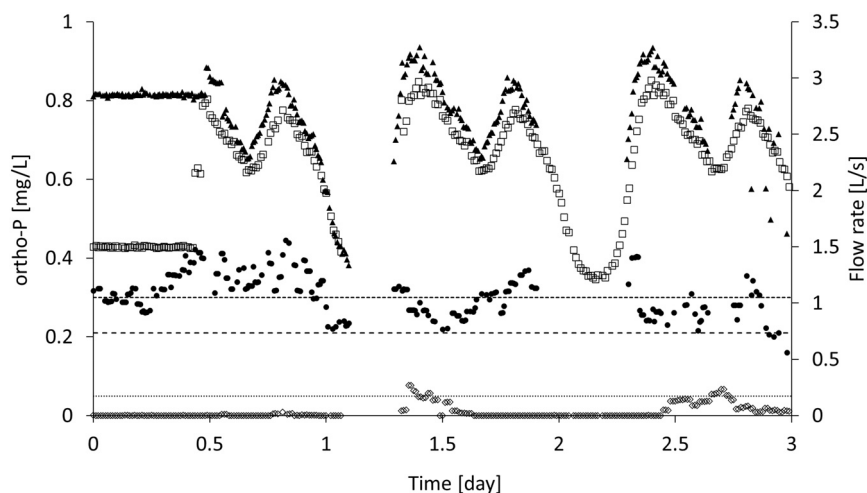


Fig. 6. Impact of diurnal flow profile on the effluent P for the pile cloth media filter (CF) (●) and ballasted flocculation (BF) (◇) with flow rates for CF (▲) and BF (◆). TP target (---●), ortho-phosphate target (----) and P detection limit (.....).

The final tests exposed both technologies to diurnal flow operation (Fig. 6). As before, the BF system demonstrated a greater robustness to the changes with only minor increases in effluent phosphorus concentration occurring at two points leading to peaks of 0.07 and 0.08 mg P/L. These coincided with the largest variations in flow and indicate that the level of change poses the greatest risk. In the case of the CF the levels

fluctuated between 0.16 and 0.44 mg P/L although the peaks in concentration did not necessarily coincide with the peaks in flow. Interestingly, the largest changes in flow did not have the same impact as seen for the BF system and mostly indicating again that the CF is less sensitive to flow variation. However, the effluent concentration was higher and fluctuated more than the BF indicating that the systems

require further optimisation to consistently deliver sub 0.3 mg P/L effluents and this is likely related to the pre-flocculation system. The flow variations during this test may have impacted floc formation and breakage through changes in retention time and velocity in the flocculation tanks. This can be supported through previously reported trials of a 10 µm microsieve technology that achieved very ultralow effluent P concentrations (Langer and Scherman, 2013). This system compared ferric and aluminium coagulants in combination with polymer dosing to reduce a wastewater phosphorus concentration from approximately 0.5 mg P/L as total phosphorus down to 60 µg P/L.

Ultimately, the results provide a critical insight for technology selection for the industry. Indeed, when it comes to implementation in full-scale not only performance, but also capital and operational costs will be considered. A previously published life cycle assessment for tertiary P removal technologies revealed that energy demand increases when moving from high-rate sedimentation over a microsieve to UF (Remy et al., 2014). Translated to this study, this means that ballasted flocculation (i.e. high-rate sedimentation) can be a sensible choice for medium-to-large wastewater treatment plants where effluent consents below <0.3 mg P/L are set. The cloth filtration technology can be implemented at smaller sites that have consents between 0.3 and 0.5 mg P/L. Based on this work and projected costs, the ultrafiltration technology will likely not be a suitable solution at this stage because it still requires more understanding into impacts of coagulant types and up-stream flocculation to achieve resilient performance at conservative chemical consumption. To deliver the required effluent P concentrations, coagulant doses at minimum molar ratios of 1.37 Fe:P were shown to be sufficient at short contact times as has been seen for the ballasted coagulation process. Moreover, cloth filtration and ultrafiltration were observed to have limitations which were not only influenced by coagulant doses but also coagulant types and up-stream flocculation conditions.

3.5. Comparative analysis

In summary, all systems tested performed well and were able to reduce the phosphorus down to low levels. The SF and BF performed better as they were able to robustly achieve effluent residuals of 0.1 mgP/L while the CF and MF could only achieve levels of 0.3 mgP/L sustainably (Table 3). In addition, the CF required higher coagulant doses than the MF and BF to achieve the lowest levels. However, it should be noted that the BF relied on additional polymer dosing for flocculation as part of the process. Effluent quality and the associated chemical use are critical factors to consider when developing a full-scale scheme, but technology selection will depend on the specific requirements of each case with other factors such as capital and operational costs coming into play. The findings from this study hence provide valuable insights and guide for the implementation of these technologies. Variations in the water characteristics influenced the systems

Table 3
Comparative evaluation of the technologies for all conditions tested.

	Sand filtration	Pile cloth media filtration	Membrane filtration	Ballasted flocculation
Robustness at steady state	+++	++	++	+++
Lowest residual achievable (mgP/L)	0.1	0.3	0.3	0.1
Chemical use	/	++	+++	++
Impact of influent characteristics	/	P: ↓ SS: =	P: = SS: =	P: = SS: ↓
Impact of flow variations	/	↓	/	=

+++; very good; ++; good; /; not tested; P: phosphorus; SS: suspended solids; ↓; performance decrease; =; performance stable.

differently. Indeed, the MF and BF maintained high performance with variations in the inlet phosphorus while the CF performance decreased with increasing P. Alternatively, increased solids did not affect the CF and MF but reduced the performance of the BF. Also, flow variations through either storms or diurnal flow profiles affected the CF's treatment performance as opposed to the BF. These results highlight that stability in flow and characteristics of the water to be treated is critical for stable operation and performance of the pile cloth media filter.

Some of the difference observed can be related to the differences in processes involved. While all systems relied on coagulation to transfer the phosphorus from soluble to particulate, the BF benefited from additional flocculation with the polymer and ballast, leading to better aggregation of smaller particles into larger, stronger flocs which in turn can also help remove other particles by sweep coagulation. For the other systems, treatment performance will be dictated by the performance of both the coagulation and the separation process. Interestingly, the CF and SF have been reported to achieve similar complete removal of particles over 100 µm with then decreasing efficiencies for smaller particles (Kaminsky et al., 1997; Naddeo and Belgiorno, 2007; Sembiring et al., 2021; Socionovo Gioacchini, 2020). However, SF have also been reported to be efficient at removing very small particles (< 1 µm) through Brownian motion and interception (Zamani and Maini, 2009). This may explain the better performance generally observed here for the SF over the CF. The MF relies on a very small pore size at 0.03 µm to remove even the smallest of particles. In this case, the poorer performance observed, when compared to the BF and SF, could be due to an under-optimised coagulation process, highlighting again the importance of the optimisation of this stage to ensure high performance.

4. Conclusions

The current study provides a unique side by side assessment of technologies integrating coagulation with different separation processes and new insights in the key attributes of these technologies for the removal of phosphorus to sub 1 mg P/L. In particular, it clearly establishes how the choice for solids retention technologies can be decisive to achieve effluent P below 0.3 mg P/L which can be used for technology selection for full scale applications.

The benchmark system including coagulation followed by sand filtration, was shown to be very robust at steady state. From the three technologies tested, the ballasted coagulation process was found to perform most consistently during steady state and dynamic operations, reliably reaching effluent P values below the ortho-P target of 0.21 mg P/L. It was in fact demonstrated to be able to meet ortho-P targets of sub 0.1 mg P/L. However, in tests with a high solids feed, the ballasted coagulation system was outperformed by the membrane and it is assumed that the higher solids content resulted in larger, more fragile flocs that were prone to breakage in the ballasted system, operated with high mixing rates to maintain the ballasting agent in suspension.

Pile cloth media filtration demonstrated limitations to meet the sub 0.3 mg P/L effluent target but was found to be robust at meeting sub 0.5 mg P/L, highlighting its place for application when less strict consents are required. Its performance was improved performance with the high solids feed showing that the formation of larger flocs help improve the separation in the cloth. The pile cloth media filtration was impacted by an increased influent P concentration but dealt well with variations in flows.

The membrane displayed poor robustness during the steady state trial with the production of a broad range of effluent qualities. However, the membrane was found to deliver excellent performance when fed with the effluents with increased P or high solids owing to its ability to equally remove the larger solids as well as the smaller fragments from floc breakage. Both the pile cloth media filtration and ultrafiltration membrane were able to achieve the effluent target though at higher coagulant doses. Finally, the upstream coagulation and flocculation system was shown to be an important factor and its optimisation is

critical for optimum performance.

CRedit authorship contribution statement

Olga Murujew: Writing – original draft, Methodology, Investigation, Formal analysis, Data curation. **Andrea Wilson:** Writing – review & editing, Methodology, Investigation, Conceptualization. **Peter Vale:** Writing – review & editing, Methodology, Investigation, Conceptualization. **Yadira Bajón-Fernández:** Writing – review & editing, Investigation, Funding acquisition, Conceptualization. **Bruce Jefferson:** Writing – review & editing, Validation, Supervision, Methodology, Investigation, Funding acquisition, Conceptualization. **Marc Pidou:** Writing – review & editing, Validation, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.scitotenv.2025.179170>.

Data availability

Data will be made available on request.

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Robustness and resilience of different solid-liquid separation technologies for tertiary phosphorus removal to low levels by coagulation

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