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Effects of wastewater pre-treatment on clogging of an intermittent sand filter

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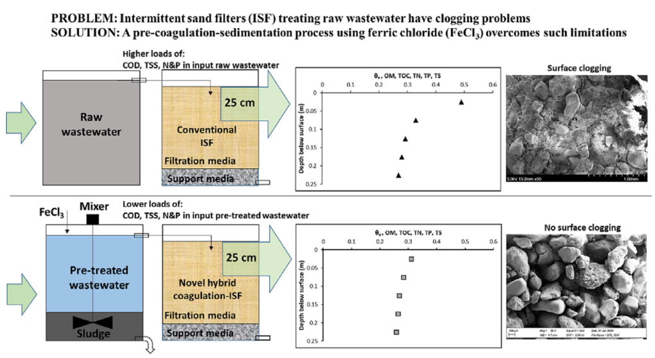
HIGHLIGHTS

- A pre-treatment coagulation step by $FeCl_3$ improves filter efficacy.
- The conventional ISF clogged and lost 85 % of its initial infiltration capacity.
- $FeCl_3$ prevented clogging and contributed to 45 % of clogging alleviation.
- Organic matter and suspended solids accumulation are the main causes of clogging.
- There were strong internal relationships between clogging indicators.

GRAPHICAL ABSTRACT



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ABSTRACT

Intermittent sand filters (ISFs) are widely used in rural areas to treat domestic and dilute agricultural wastewater due to their simplicity, efficacy and relative low cost. However, filter clogging reduces their operational lifetime and sustainability. To reduce the potential of filter clogging, this study examined pre-treatment of dairy wastewater (DWW) by coagulation with ferric chloride ($FeCl_3$) prior to treatment in replicated, pilot-scale ISFs. Over the study duration and at the end of the study, the extent of clogging across hybrid coagulation-ISFs was quantified, and the results were compared to ISFs treating raw DWW without a coagulation pre-treatment, but otherwise operated under the same conditions. During operation, ISFs receiving raw DWW recorded higher volumetric moisture content (θ_v) than ISFs treating pre-treated DWW, which indicated that biomass growth and clogging rate was higher in ISFs treating raw DWW, which were fully clogged after 280 days of operation. The hybrid coagulation-ISFs remained fully operational until the end of the study. Examination of the field-saturated hydraulic conductivity (K_{fs}) showed that ISFs treating raw DWW lost approximately 85 % of their infiltration capacity in the uppermost layer due to biomass build-up versus 40 % loss for hybrid coagulation-ISFs. Furthermore, loss on ignition (LOI) results indicated that conventional ISFs developed five times the organic matter (OM) in the uppermost layer compared to ISFs treating pre-treated DWW. Similar trends were observed for phosphorus, nitrogen and sulphur, where proportionally higher values were observed for raw DWW ISFs than pre-treated DWW ISFs, with values decreasing with depth. Scanning electron microscopy (SEM) showed a clogging biofilm layer on the surface of raw DWW ISFs, while pre-treated ISFs maintained distinguishable sand grains on the surface. Overall, hybrid coagulation-ISFs are likely to sustain infiltration capacity for a longer period than filters treating raw wastewater; therefore, requiring smaller surface area for treatment and minimal maintenance.

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1. Introduction

Intermittent sand filters (ISFs) are onsite wastewater treatment systems, commonly used in wastewater remediation, to remove pollutants by physical, chemical and biological mechanisms (Sylla et al., 2020). Intermittent dosing enables bacterial growth and filter aeration between doses, and hence facilitates both aerobic and anoxic metabolisms (Murnane et al., 2016). The applications of ISFs to treat domestic and septic tank effluent (Gill et al., 2009; Rodgers et al., 2011) and agricultural dairy wastewater (DWW) has been shown to be cost-effective and efficient at removing contaminants (Rodgers et al., 2005; Mohamad et al., 2022). Well-designed ISFs can achieve substantial reductions of total suspended solids (TSS), biochemical/chemical oxygen demand (BOD, COD), *Escherichia coli* (*E. coli*) and viruses (Healy et al., 2007; Torrens et al., 2009a).

Nevertheless, similar to other permeable media-based wastewater treatment systems, regular clogging is a major inherent operational challenge for ISFs (de Matos et al., 2018; Wang et al., 2021; Wu et al., 2022). Rodgers et al. (2004) reported multiple clogging events in ISFs treating DWW at different organic and hydraulic loading rates. Torrens et al. (2009b) also reported filters clogging when the system operation did not follow the recommended feeding and resting periods for ISFs that were used to treat pond effluent at an organic loading rate (OLR) ranging from 17 to 170 g COD m^{-2} d^{-1}. Clogging is usually attributed to diminished permeability and infiltration capacity caused by surface or interstitial deposits of TSS present in the influent wastewater, or porosity reduction from accumulation of bacterial biomass and production of hydrated extracellular polymers (exopolymers) within the matrix of the sand (Leverenz et al., 2009). However, ISF clogging has been predominantly regarded as a surface sealing phenomenon (Rodgers et al., 2004). Factors such as the loading regime, organic and hydraulic loading rates (HLRs) of the applied wastewater, and the filter media properties, impact distribution of the treatment zone in the filter media and dictate the depth of biofilm accumulation in the filter media.

Clogging becomes apparent in ISFs when surface ponding occurs and the effluent flowrate declines (Knowles et al., 2011). From a technical perspective, clogging of filters may be monitored and quantified in a number of ways. During operation, the head loss method is commonly used to determine the occurrence and extent of clogging in continuously operated systems (i.e., rapid and slow sand filters; Mesquita et al., 2012; De Souza et al., 2021), while monitoring of the volumetric moisture content method (θ_v) is more suitable for intermittently loaded systems such as ISFs (Rodgers et al., 2004; Ruane et al., 2014). Measuring the outflow rate and infiltration time for one dose using tracer tests can be used as a non-destructive method to monitor clogging in ISFs during operation (Nivala et al., 2012; Gikas et al., 2017). As clogging developed in the filter, the infiltration time for one dose increased with time. Following the occurrence of surface ponding and the subsequent destructive sampling of the filter, field-saturated hydraulic conductivity (K_{fs}) is the best indicator to measure the development of clogging (Rodgers et al., 2004; Lianfang et al., 2009; Le Coustumer et al., 2012). As the filter clogs over time, θ_v increases and K_{fs} decreases (Knowles et al., 2011; Ruane et al., 2014). Depending on the permeability of the investigated media, measurement of K_{fs} can be conducted using either a constant head test (BSI, 1990a) or falling head test (ASTM, 2010). Other common procedures of investigation include organic and biomass content estimation through loss on ignition (LOI), chemical analysis of the filter media at different depths, biomass layer visualization via scanning electron microscopy (SEM), and X-Ray diffraction (XRD) technique (Pedescoll et al., 2009; Knowles et al., 2011; Grace et al., 2016).

There are many measures which can be applied to prevent and delay clogging in ISFs. Suspended solids and particulate COD can be controlled and eliminated by engineering methods such as coarse filtration and pre-sedimentation (de Matos et al., 2018). Biomass development can be managed by system resting (Torrens et al., 2009b), surface scraping (De Souza et al., 2021) and the use of earthworms (Wang et al., 2010; Singh et al., 2018). Pre-treatment of wastewater, lowering hydraulic and organic loading rates and decreasing dosing frequency have also been found to result in both enhanced performance and extended operational periods without clogging (Leverenz et al., 2009; Chen et al., 2021a, 2021b), though research in this area is limited.

A pre-treatment step prior to ISFs is recommended to reduce the concentration of the applied wastewater and prevent premature clogging (Healy et al., 2007). Pre-treatment of wastewater by a mixture of coagulation and sedimentation may address the shortcomings of ISFs and reduce their inherent operational problems. Cameron and Di (2019) obtained a high removal of organic matter (OM), TSS and nutrients (nitrogen (N) and phosphorus (P)) for DWW clarified with a ferric-based coagulant at small doses. Therefore, this method can theoretically reduce the size of a subsequent ISF system, and/or increase its operational period prior to clogging. Selection of a suitable type of chemical coagulant, pH, dosage rate and mixing power/time are important design parameters to control and optimize the coagulation-flocculation process and ensure better wastewater purification (Karam et al., 2021). While there are many available coagulants that can be used for DWW treatment, Mohamed et al. (2020) evaluated aluminium sulphate ($Al_2(SO_4)_3$), poly-aluminium chloride and ferric chloride ($FeCl_3$) for DWW coagulation, and found that $FeCl_3$ was the optimum coagulant for this application. Their study took into account many aspects to appraise and rank these chemical coagulants such as treatment efficacy, treatment cost and sludge production quantities. Ferric chloride has been widely used for municipal and industrial wastewater treatment and been shown to be very effective at contaminant removal (El Samrani et al., 2004; Guerreiro et al., 2020). In addition, $FeCl_3$ demonstrated effective fouling mitigation and clogging alleviation in membrane-based biological wastewater treatment systems (Dong et al., 2015; Tang et al., 2018).

Nevertheless, to date, no study has investigated the integration of $FeCl_3$ coagulation with ISF treatment of DWW to alleviate clogging for pilot-scale DWW treatment. Therefore, the aims of this study were, for the first time, to (1) use $FeCl_3$ in a coagulation-sedimentation process as a pre-treatment step for ISFs treating DWW over the duration of a full milking season, and (2) ascertain feasibility of this approach for improved performance of ISF system by comparing indicators of clogging for this hybrid system to conventional ISFs (without pre-treatment), using a range of physical and chemical analyses.

2. Materials and method

2.1. Experimental set-up

Six outdoor pilot-scale intermittent sand filters for treating raw ($n = 3$) and pre-treated ($n = 3$) DWW were operated (in 2021) for a period of 301 days, in single-pass operation mode (Fig. 1). All filters were 0.9 m deep and 0.5 m in diameter, and were designed following US EPA guidelines (USEPA, 1980). In each filter, a 0.1 m layer of distribution gravel (10–14 mm in size) overlaid a 0.65 m layer of fine sand media (effective size, $d_{10} = 0.17$ mm; uniformity coefficient, UC = 2). To prevent washout of the filter media, the bottom layer of sand was underlain by a 0.1 m layer of pea gravel (10–14 mm in size).

Raw DWW was collected weekly in a 1000 L capacity intermediate bulk container (IBC; Fig. 1) from Teagasc Moorepark Dairy farm, Fermoy, Co. Cork, Ireland. The DWW comprised washings from the milking parlour and collecting yard, and from cleaning the milking plant. Following this, 200 L of raw DWW was decanted from the IBC tank and transferred into another storage container (hereafter referred to as raw DWW; Fig. 1-A). The remaining raw DWW in the IBC tank (800 L) was mixed and treated with $FeCl_3$ solution at a dosage of 440 mg Fe L^{-1} of DWW (10.35 g Fe g^{-1} P). Mohamed et al. (2020) showed that this dosage was optimal for the removal of contaminants (COD, TSS, turbidity TP, TN, *E. coli*) present in DWW. The mixture was then allowed to settle for 3 h, after which 200 L of the supernatant was decanted into another storage container (hereafter referred to as pre-treated DWW; Fig. 1-B). Raw and pre-

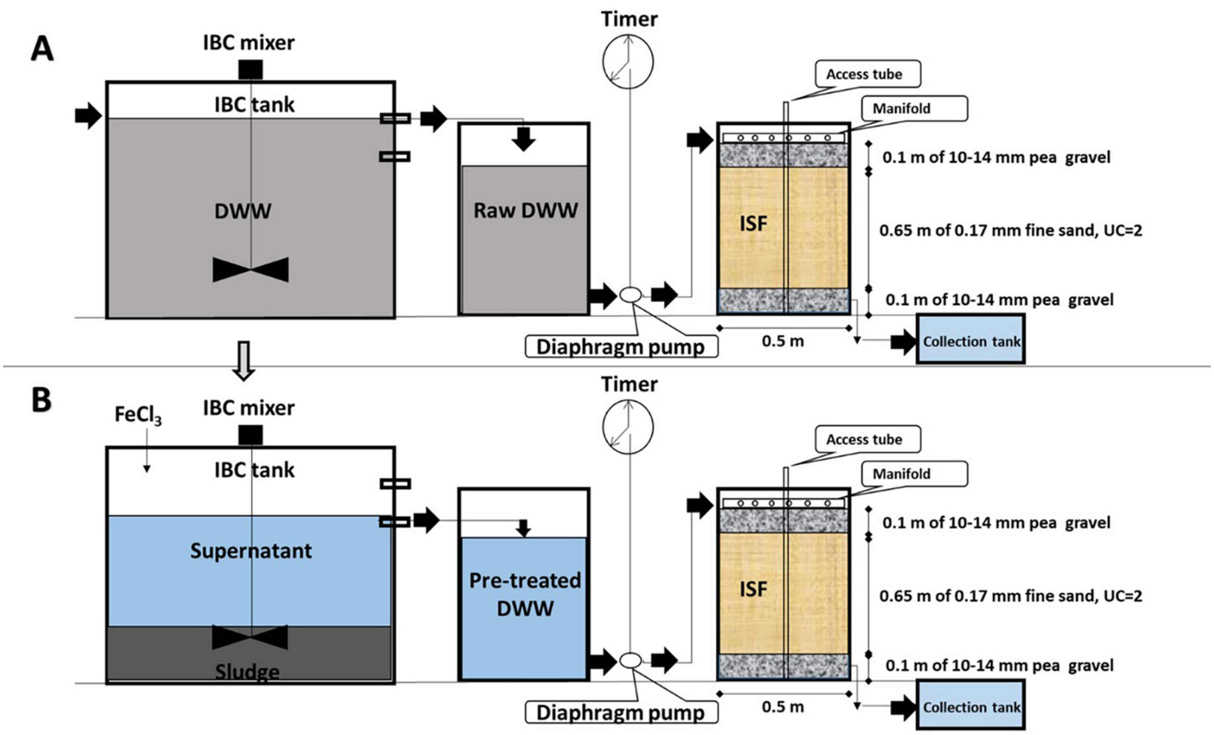

Fig. 1. Experimental set-up showing schematic views of the (A) raw DWW system and (B) pre-treated DWW system.

treated DWW were made up weekly and pumped intermittently from the storage containers onto the replicated ($n = 3$) single-layer sand filters, using a diaphragm pump controlled by electronic timers (Fig. 1). The wastewater in the storage containers were agitated regularly to ensure homogeneity. The wastewater was distributed over the filter media using un-plasticised polyvinyl chloride (uPVC) distribution manifolds (Fig. 1). The experiment spanned the entire 2021 milking season and consisted of four phases of different organic and hydraulic loading rates (Table 1). The filters were operated with the same OLR in the first and second phases, and the same HLR in the third and fourth phases (Table 1). Surface ponding on the raw DWW filters occurred at day 280 of operation (middle of Phase 4), so hydraulic loading was discontinued for those filters.

2.2. Analysis

2.2.1. Water quality parameters

Raw DWW and pre-treated DWW samples were collected and analysed weekly. Turbidity was measured using an Orion AQUAfast turbidity meter (ThermoFisher Scientific, USA). COD was measured using the dichromate digestion method with HACH testing kits (HACH, USA). Total suspended solids were measured using the gravimetrical method by filtering the samples through a Whatman GF/C (pore size of filters = 1.2 μm) using a vacuum pump, and drying the filter paper for 2 h at 103–105 °C. Total phosphorus (TP) and total nitrogen (TN) were measured using the Persulphate Digestion/Oxidative method through (HACH Ganimede instruments). All tests were carried out in accordance with standard methods (APHA, 2005).

2.2.2. Clogging detection methods

pt? >During operation, clogging in filters was monitored physically by measuring volumetric moisture content of sand layers. As the build-up of biomass on the ISF increases with time, the filters retain more water between sand grain/pores, increasing the θ_v and reducing the infiltration capacity/ K_{fs}. At the end of the experiment, all filters were dismantled, and the physical and chemical properties of the sand were characterised in 0.05 m increments to a total depth of 0.25 m below the sand surface. The physical clogging at the end of the study was measured by determining the hydraulic conductivity (infiltration capacity) of sand layers. The chemical indicators, which comprised carbon (C) /OM, N, P and sulphur (S), were measured at the end of the study as an indication of bacterial biomass development in the filters.

Table 1
Experimental phases of different operational regimes of OLR, total suspended solids loading rate (TSSLR) and HLR applied to raw and pre-treated DWW filters during a period of 301 days (Mohamed et al., 2022).

Operation mode	Phase	Days of operation	Waste source	OLR (g COD m^{-2} d^{-1}) Mean ± SD	TSSLR (g m^{-2} d^{-1}) Mean ± SD	HLR (L m^{-2} d^{-1}) Mean ± SD	Dosing frequency/day
Same OLR	1	49	Raw	30 ± 6	15.7 ± 3	6 ± 1.5	4
			Pre-treated	30 ± 6	2.8 ± 0.5	20 ± 4	
	2	154	Raw	15 ± 5	5.1 ± 1.5	3 ± 0.8	4
			Pre-treated	15 ± 5	0.65 ± 0.2	10 ± 2	
Same HLR	3	42	Raw	55 ± 8	18 ± 3	10 ± 2	4
			Pre-treated	15 ± 5	1.1 ± 0.3	10 ± 2	
	4[a]	56	Raw	110 ± 10	46 ± 5	20 ± 4	8
			Pre-treated	30 ± 6	4 ± 0.8	20 ± 4	

[a] Ponding occurred for raw DWW filters in Phase 4.

2.2.2.1. Physical properties. From day 70 (Phase 2), when the filters were fully biologically active, the build-up of biomass in the filters was measured, by proxy, by time-domain reflectometry (TDR) (Rodgers et al., 2004). The sand filters were instrumented with 1 m-deep access tubes (Fig. 1; type ATL1, Delta-T Devices Ltd., Cambridge, UK) to allow the θ_v to be measured at different depths using a TDR probe (PR1/6d-02, Delta-T Devices Ltd., Cambridge, UK). In order to monitor the biomass build-up, the θ_v was recorded weekly at each 0.05 m depth increment to a total depth of 0.25 m below the top of the sand. Readings were taken in millivolts using a voltmeter (type HH2, Delta-T Devices Ltd., Cambridge, UK) and were converted to units of $m^3\ m^{-3}$ using the manufacturer's calibration curve.

At the end of experiment, six intact sand cores, 0.05 m in diameter (representing 6 % of the total surface area), were extracted at each 0.05 m incremental depth below the surface and used to determine the K_{fs} (m s^{-1}) of each layer by the constant head method (BSI, 1990a). In this method, the intact sand cores, in open-ended pipes, were subjected to a constant ponded head of water, z. The constant head was maintained in each sand core by an overflow pipe. Flow rates (Q; $m^3\ s^{-1}$) were measured by graduated cylinders, positioned under the open-ended pipes. A virgin sand specimen was used to compare the reduction in K_{fs} with depth for both sets of filters (raw and pre-treated DWW). The K_{fs} was calculated using Darcy's empirical law (Eq. (1))

$$Q = A * K_{fs} * \left(1 + \frac{z}{l}\right) \quad (1)$$

where Q $_{is}$ the flow rate, A is the cross-sectional area (0.002 m^2), z is the water depth (0.05 m), and l is the height of sand core (0.05 m).

2.2.2.2. Chemical properties. Following deconstruction of the filters, each 0.05 m layer below the surface was analysed for a variety of parameters. Organic matter was measured using the LOI technique by drying samples at 105 °C, and then ashing at temperatures of 430 °C (BSI, 1990b). This method can give an indication of biomass distribution within the filter (a physical mechanism responsible for clogging). Total nitrogen was measured using the Dumas Technique (Method 949.12, AOAC, 1990). Total phosphorus and total sulphur (TS) were measured using hydrochloric and nitric acid (aqua-regia) digestion methods (SW 486 Method 3050B, USEPA, 1996). Total organic carbon (TOC) was measured using the DUMAS combustion method (BS EN 15936, BSI, 2012). As there was a strong relationship between OM and TOC (OM/TOC = 2.2, R^2 = 0.99), measured values of TOC were used to estimate OM for values that were below the detection limit (Schumacher, 2002).

2.2.2.3. Microscopic visualization. Scanning electron microscopy was used to view the biomass build-up on individual sand grains at the surface of raw and pre-treated DWW filters, as well as on virgin sand samples. Intact samples were taken from the surface of the filters. The structural integrity of the biofilms on the sand were preserved with adequate primary fixation in paraformaldehyde and glutaraldehyde, followed by gradual dehydration (using ethanol: 30 %, 50 %, 70 %, 90 % and 100 %) and critical point drying. When dried, the samples were mounted onto aluminium stubs with a double-sided sticky tab and gold sputter coated (Q150R ES plus, Quorum, Sussex, UK), and were viewed with a scanning electron microscope (Model S4700, Hitachi, Tokyo, Japan) at 50× magnification.

2.3. Data analysis

Statistical analyses were carried out using SAS 9.4 (SAS Institute Inc., USA). Differences in physical and chemical properties between raw DWW and pre-treated DWW filters were analysed using PROC MIXED model. PROC MIXED addressed challenges associated with non-normal distribution. The model was designed as a two-factor factorial experiment (2*5) with three replications, consisting of two categorical independent variables: Treatment (two treatments: raw DWW, pre-treated DWW), Depth (five depths: 0–0.05 m, 0.05–0.1 m, 0.1–0.15 m, 0.15–0.2 m, 0.2–0.25 m). The main effect of each factor, along with interaction effect (Treatment × Depth), were investigated by the model against each physical and chemical parameter, which was set as a continuous dependent variable in the model. LSMEANS statement (with a Tukey adjustment) identified where significant differences occurred between raw and pre-treated DWW filters at specific depths. For the volumetric moisture content parameter, the model incorporated an additional factor: Week (multiple weeks that varied from phase to phase), beside the interaction between the factors (Treatment × Depth* Week) as fixed terms. Three separate models were constructed for θ_v, a separate model for each phase of the experiment, to account for methodological differences between phases as described in Table 1. Probability values of $p > 0.05$ were deemed not to be significant.

3. Results and discussion

3.1. Impact of ferric chloride pre-treatment on water quality parameters

The deployment of $FeCl_3$ flocculant for the clarification and treatment of DWW reduced COD significantly, with an average decrease of 75 % ($p < 0.001$; Fig. 2). This finding was consistent with the study of Mohamed et al. (2020), who attained an 85 % reduction in COD for DWW amended with $FeCl_3$ at a similar dosage. The removal of the particulate COD fraction was the main mechanism of COD reduction by $FeCl_3$ (Mohamed et al., 2020).

The significant reductions ($p < 0.001$) in turbidity, TSS and TP (an average decrease of >95 %; Fig. 2) by $FeCl_3$ were comparable to those obtained by Mohamed et al. (2020) and Cameron and Di (2019), who used $FeCl_3$ and poly-ferric sulphate flocculants to clarify DWW at optimal dosages of 470 and 214 mg Fe L^{-1}, respectively. Ferric chloride removed turbidity and TSS primarily through destabilization of colloidal particles/SS or so-called hydrolysis (sedimentation process of $Fe(OH)_3\downarrow$), while the chemical precipitation in the form of ferric phosphate bonds ($FePO4\downarrow$) was the main mechanism of P elimination (Bratby, 2016).

Total nitrogen was also reduced by $FeCl_3$ flocculant (an average decrease of 46 %; $p < 0.001$; Fig. 2), but the reduction was lower than the reductions of COD, TSS, turbidity and TP. Particulate N removal through sedimentation was the main mechanism of TN removal by $FeCl_3$ (Mohamad et al., 2022). The residual N in the treated DWW comprised mainly soluble forms of N such as dissolved organic nitrogen (DON) and ammonium (NH_4-N) (Mohamed et al., 2022), which can be only eliminated and reduced through other chemical and biological transformation mechanisms such as bio-adsorption, nitrification-denitrification and volatilization (Chen et al., 2020). Similarly, Cameron and Di (2019) and Mohamed et al. (2020) reported maximum TN removals of 57 % and 35 %, respectively, using Fe-based coagulants.

3.2. Clogging indicators

3.2.1. Physical indicators

Increasing hydraulic and organic loading rates during the operation of the filters produced significantly higher θ_v in the uppermost layers of the filters, which was indicative of the potential for clogging in these layers. In each phase, there were significant depth, treatment and depth*treatment effects on θ_v (Table S1). In all cases, the θ_v reduced significantly ($p < 0.001$) with depth from the sand surface. During Phase 2, there was no significant difference ($p > 0.05$) in the θ_v between raw and pre-treated DWW filters at each depth analysed (Table S1; Fig. 3-A). These results indicate that there was no difference in the biomass build-up between treatments, when the filters were operated at the same OLR.

In Phase 3, once the HLR of the raw DWW filters was increased to that of the pre-treated DWW filters, the θ_v of the uppermost sand layer (0–0.05 m) increased by 50 %, significantly higher than that attained by pre-treated DWW filters ($p < 0.001$; Fig. 3-B). The differences in θ_v between raw and pre-treated DWW filters reduced with depth beneath the filter surface

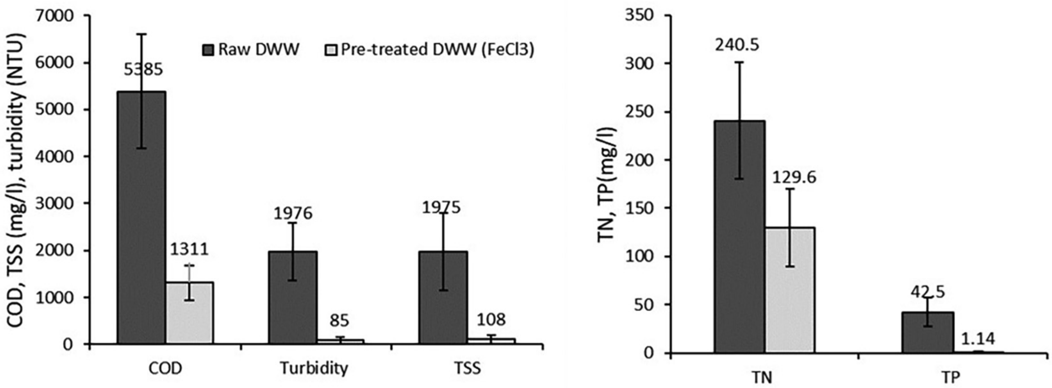

Fig. 2. Raw DWW and pre-treated DWW characteristics for COD, total suspended solids (TSS), turbidity, total nitrogen (TN) and total phosphorus (TP).

(Fig. 3-B), and there were no significant differences in θ_v at deeper depth increments (0.1–0.25 m; Table S1).

In Phase 4, the θ_v for the uppermost sand layer (0–0.05 m) of the raw DWW filters was significantly ($p < 0.001$) higher than that obtained by pre-treated DWW filters (Fig. 3-C). Rodgers et al. (2005) studied a stratified ISFs loaded for 342 days with synthetic DWW at a HLR of 20 L m^{-2} day^{-1} and an OLR of about 25 g COD m^{-2} day^{-1} and found that the θ_v increased to a maximum value of approximately 0.4 m^3 m^{-3} at the uppermost sand layer. This value was similar to that achieved by the pre-treated DWW filters in the current study, which were operated almost in the same conditions (HLR of 20 L m^{-2} d^{-1}, OLR of 30 g COD m^{-2} day^{-1}). There was also a significant difference between the θ_v in the raw and pre-treated DWW filters for the deeper depth increments, except for the deepest monitored layer (0.2–0.25 m; Fig. 3-C; Table S1), indicating that biomass accumulation due to the increased OLR in the raw DWW filters had abated by that depth. The highest θ_v values observed in raw DWW filters were similar to previous literature findings of ISFs that did not incorporate a pre-treatment step for DWW treatment (Rodgers et al., 2004). The $FeCl_3$ reduced the OLR significantly in pre-treated DWW filters (Table 1), contributing to significantly lower θ_v values (at most depths) in pre-treated DWW filters relative to raw DWW filters, although both sets of filters were operated under the same HLR. These results indicate that pre-treatment by $FeCl_3$ increases the operational longevity of an ISF, allowing for higher HLR operation or lower size footprint than the conventional ISF.

There were significant depth and treatment*depth effects on K_{fs} (Table S1). At the end of experiment, the raw DWW filters lost about 85 % of their initial K_{fs} (i.e. $K_{virgin\ sand}$) in the uppermost layer (0–0.05 m; Fig. 4), likely due to biomass development. The decline in the relative K_{fs} value for raw DWW filters in the current study was comparable to that of other studies. Rodgers et al. (2004) measured a reduction of 98 % in the initial K_{fs} for the uppermost layer of an ISF treating synthetic DWW, and Schwager and Boller (1997) also observed a reduction >95 % in the initial K_{fs} for the uppermost 0.04 m layer for ISFs treating septic tank effluent at a HLR of 120 L m^{-2} d^{-1}. Interestingly, due to the $FeCl_3$ pre-treatment, the pre-treated DWW filters only had a 40 % loss of their initial K_{fs} in the uppermost layer (0–0.05 m; Fig. 4), which was significantly lower than raw DWW filters ($p < 0.01$).

Ponding occurred on day 280 in all raw DWW filters, while pre-treated DWW filters maintained a higher infiltration rate than the applied HLR. Although the study was operated for 302 days, it is clear from the K_{fs} measurements that the $FeCl_3$ prevented clogging and contributed to 45 % of ISF clogging mitigation. Therefore, longer operation is expected for the pre-treated filters. As both sets of filters were operated with the same HLR, but different OLRs in Phase 4 (110 versus 30 g COD m^{-2} d^{-1} for the raw and pre-treated filters; Table 1), it is likely that clogging occurred due to the increased organic matter and sediment contained in the raw DWW. Besides TSS capture, the main mechanism responsible for sand clogging on raw DWW filters was secretion and biomass accumulation in the uppermost layer of ISFs. The increase in the biomass in the uppermost layer, and TSS deposition, decreased the pores size and therefore reduced the K_{fs} (Fig. 4), decreasing infiltration in the uppermost layer, increasing the θ_v (Fig. 3). In the deeper layers, there was no significant differences ($p > 0.05$) in the K_{fs} between raw and pre-treated DWW filters (Table S1; Fig. 4). The reduction in K_{fs} diminished with depth below the filter surface until the K_{fs} returned to that of the virgin sand at a depth of 0.2–0.25 cm (Fig. 4). A similar trend was also observed by Grace et al. (2016), who used ISFs (d_{10} = 0.18 mm, UC = 2.19, column depth = 1 m) to treat synthetic wastewater for 90 days. This implies that removing the upper layer to a depth of approximately 0.2 m below the surface will fully restore the filter.

3.2.2. Chemical indicators

Trends similar to the volumetric water contents were observed for the chemical properties (OM, TP, and TS): significantly higher values for the raw DWW than the pre-treated DWW, with values decreasing with depth below the sand surface. There were significant effects of treatment, depth, treatment*depth on all chemical parameters (OM, TP, TS; Table S2; $p < 0.001$).

The average OM in the uppermost layer (0–0.05 m) for raw DWW filters was significantly higher ($p < 0.001$) than pre-treated DWW filters (Fig. 5-A), indicating that biomass build-up (i.e., OM content in filter relative to OM content in virgin sand) in the raw DWW filters was more than fivefold the biomass accumulated in pre-treated DWW filters. The high OM content in raw DWW filters was similar to those reported in previous studies of ISFs that did not include a pre-treatment step. For example, Ruane et al. (2014) found the OM of the uppermost layer was more than five times the OM of virgin sand. Rodgers et al. (2004) found the OM content in the uppermost layer at clogging was more than double the OM of virgin sand, which is comparable to the raw DWW filters in the current study (Fig. 5-A). Unlike pre-treated DWW filters, the biomass build-up on raw DWW filters extended to the 0.05–0.1 m layer (Fig. 5-A). This was also evident by a colour change in the sand through these layers. Chen et al. (2021b) reported that increasing either influent strength or HLR extends the clogging development into deeper ISF layers. Applying this finding to our study, the higher concentration of contaminants (in the raw DWW compared to the pre-treated DWW) is likely to have contributed to the observed increased OM in the deeper layers of the filters.

Pre-treatment by $FeCl_3$ led to a significant reduction ($p < 0.001$) of COD, TSS, and TP loading rates on pre-treated DWW filters (Fig. 6). The cumulative COD load on raw DWW filters (2 kg) by the end of the study was twice that accumulated on pre-treated DWW filters (Fig. 6-A). Heterotrophic bacteria/ biomass utilized approximately one third of this influent COD for cell synthesis, while the remainder was used for catabolism (energy used for respiration and maintenance; Henze et al., 2008). This means that the potential biomass generated by raw DWW filters (approximately 660 g COD, or 446 g OM, COD/OM = 1.48) was approximately twice the

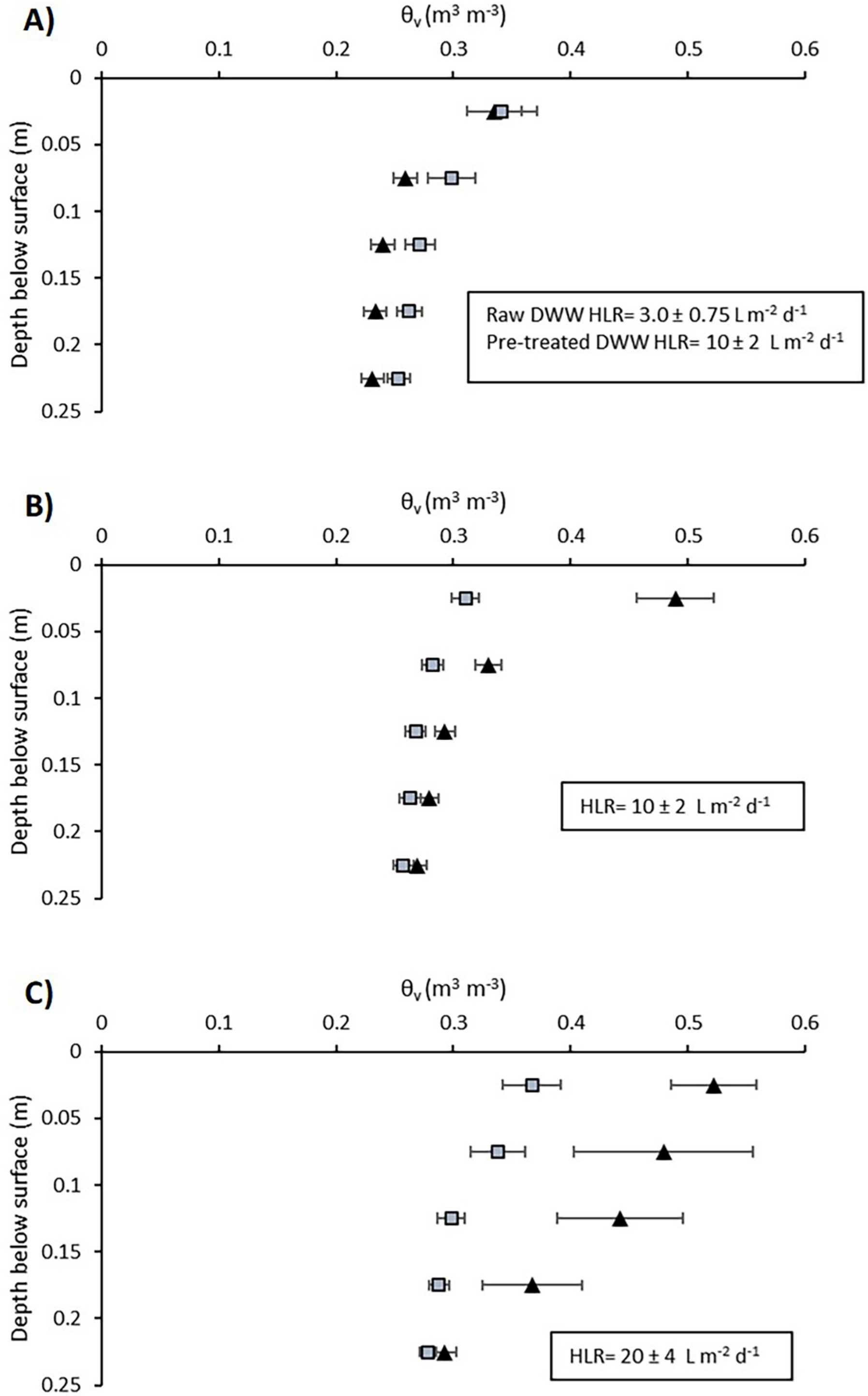

Fig. 3. Volumetric moisture contents (θ_v; mean ± SD) recorded in the filters treating raw DWW (closed triangle) and pre-treated DWW (closed box) at various depths for (A) Phase 2 (B) Phase 3, and (C) Phase 4.

biomass generated by the pre-treated DWW filters (225 g OM). This is reflected in Fig. 5-A, which indicated that the build-up of biomass on raw DWW filters (in the upper layer) was more than double that accumulated on pre-treated DWW filters. However, the estimated biomass based on the cumulative COD load was slightly higher than the actual OM presented on Fig. 5-A (assuming sand density of 2400 kg m^{-3}). This difference was expected as the influent COD was not fully removed by the filters, and some COD was released in the effluent. In addition, biomass die-off (endogenous decay) usually occurs between doses (Leverenz et al., 2009), reducing the net biomass build-up on the filters.

The cumulative TSS load on raw DWW filters (817 g) by the end of the study was eight times higher than pre-treated DWW filters (100 g; Fig. 6-B). Suspended solids in the influent wastewater, accompanied by the biomass formed through COD substrate, are the major reasons that cause clogging in ISFs (Healy et al., 2007; de Matos et al., 2018). Therefore, raw DWW filters exhibited clogging by day 280, while pre-treated DWW filters continue until the end of the study without any ponding or clogging events.

The TP in raw DWW filters was significantly ($p < 0.001$) higher than pre-treated DWW in the uppermost layer (0–0.05 m; Fig. 5-B). However, differences in TP between raw and pre-treated DWW filters reduced with

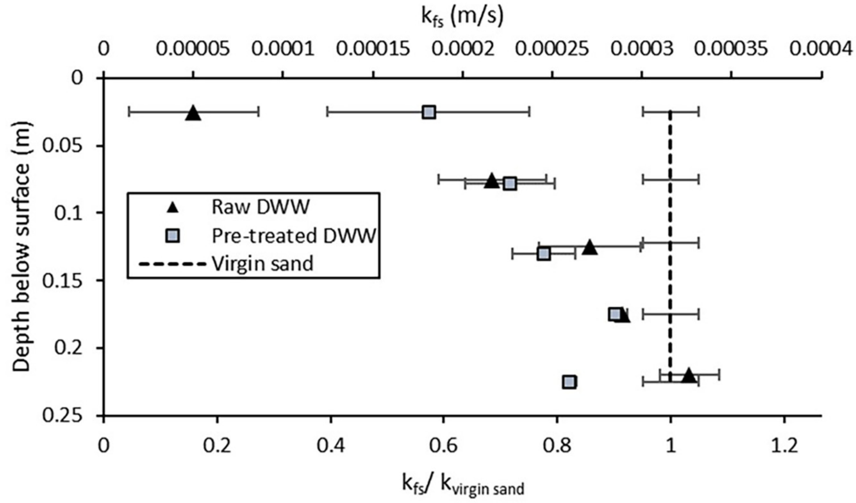

Fig. 4. Saturated hydraulic conductivity (K_{fs}; mean ± SD) measured in the filters treating raw DWW and pre-treated DWW at depths m at the end of the experiment.

the depth below the surface, with no statistical differences in the deeper layers (0.1–0.25 m; Fig. 5-B; Table S2). The TP was removed in the upstream process by $FeCl_3$ (Mohamad et al., 2022), therefore less TP was recorded in the pre-treated DWW filters. This advantage allows the pre-treated DWW filters to be operated for longer periods than raw DWW filters without P breakthrough, which is a common issue in conventional ISFs because of the limited adsorption capacity of sand (Rodgers et al., 2005; Torrens et al., 2009b; Murnane et al., 2016). The majority of the TP in DWW filters was adsorbed in the uppermost layers. Little of this TP may be used for bacterial biomass growth (typically TP in the bacterial biomass represents 3 % of OM content; Henze et al., 2008).

The cumulative TP loads on raw DWW filters (18 g) up to the end of the study was twenty times higher than pre-treated DWW filters (0.85 g; Fig. 6-C). The results were in accordance with Fig. 5-B. Assuming a typical sand density of 2400 kg m^{-3}, the mass of TP trapped within the uppermost 25 cm layer of raw DWW filters was calculated to be 13.5 g from Fig. 5-B. The missing TP can be attributed to the fact that the influent TP was not totally removed (95 % removal across the phases = 17 g).

Total nitrogen and TS retention within ISFs exhibited the same trend as TP retention. The TN in the uppermost layer of raw DWW filters (0.061 %) was more than three times the TN of pre-treated DWW filters (< 0.02 %). The TN contents in the deeper layers were below the detection limit (< 0.02 %). The TS in the uppermost layer of raw DWW filters (259 mg TS kg^{-1} sand) was significantly ($p < 0.001$) higher than pre-treated DWW filters (168 mg S kg^{-1} sand; Fig. 5-C). The difference in TS between raw and pre-treated DWW filters reduced with the depth below the surface, with no statistical differences in the deeper layers (0.1–0.25 m; Fig. 5-C; Table S2).

There was no difference in the cumulative TN load between raw and pre-treated DWW filters (Fig. 6-D). Nevertheless, the TN content in the uppermost layer of raw DWW filters was significantly higher than pre-treated DWW filters. This can be justified by two reasons: First, the influent TN applied into raw DWW filters comprised 32 % particulate N versus 3.3 % for pre-treated DWW filters. Unlike ammonium, the fate of particulate N is screening within the matrix of soil. Secondly, the heterotrophic bacterial biomass usually requires nitrogen for cell synthesis and growth (10 % of OM content; Henze et al., 2008). Since the biomass content was higher in the case of raw DWW filters, more nitrogen content was expected.

3.3. Internal relationship between clogging indicators

Fig. 7 shows clogging indicators (OM, TOC, TP, TS, θ_v) for raw DWW filters. All clogging indicators reduced with the depth below the surface (Fig. 7-A, B), except for the K_{fs}, which increased for lower layers (Fig. 7-B). This indicated that clogging reduced, and permeability increased, with depth. These results are in line with those of previous studies, which showed similar trends (Rodgers et al., 2004; Ruane et al., 2014; Grace et al., 2016). There was a linear relationship ($R^2 > 0.95$) between clogging indicators (OM, TOC, TP, TS) and filter infiltration capacity (K_{fs}) (Fig. 7-A). There was also a relationship between θ_v and K_{fs}, but the correlation was more polynomial ($R^2 = 0.97$) than linear ($R^2 = 0.72$). The clogging zone developed because of the bacterial biomass/biofilm growth at the top layers of sand filters. The bacterial biofilm could seal the porosity of sand pores/grains, and therefore could hinder and block water percolation through the filters. The bacterial biomass is made of OM and mainly consists of C, N, P and S. Therefore, as the OM content of sand increased, the infiltration capacity reduced (K_{fs}; $R^2 = 0.96$) and the moisture content of sand increased (θ_v; $R^2 = 0.6$) (Fig. 7-B). This indicates that OM can be used as an indicator to estimate infiltration capacity and clogging status at any point of filter operation. The linear relationship ($R^2 > 0.9$) between OM and other chemical parameters, indicated that the bacterial biomass (OM) consisted of 50 % C, 3.2 % P, 1.6 % S (derived from the slopes of lines; Fig. 7-B). These values were similar to the elemental compositions of bacteria reported elsewhere in the literature (Fagerbakke et al., 1996; Chen et al., 2020). Overall, depending on the available resources, any of these parameters can be used to estimate the other clogging parameters, thereby reducing the time and cost required for monitoring and analysis.

3.4. Microscopic visualization

Camera and scanning electron microscopy showed the differences in biomass build-up on the surfaces between raw and pre-treated DWW filters and their comparison to virgin sand (Fig. 8). The visual observations of biomass build-up (organic deposits) were in agreement with the measurements of θ_v, K_{fs}, and chemical properties of OM, TOC, TP and TS. In the case of virgin sand (Fig. 8-A) and pre-treated DWW filters (Fig. 8-B), the sand grains on the surface were clearly distinguishable, while biomass accumulation made the sand particles and pores indistinguishable on the surface of the raw DWW filters (Fig. 8-C), meaning that the clogging layer formed more quickly on the surface of these filters. Furthermore, Fig. 8-C indicates that the clogging zone developed as a gel-like/cake-forming layer (*schmutzdecke*) on the surface. However, the layers below the surface had distinguishable sand grains similar to Fig. 8-A and B (Fig. S1). This indicates that removing the clogging layer to a depth of approximately 0.05 m below the surface will partially restore the filter in the event of clogging.

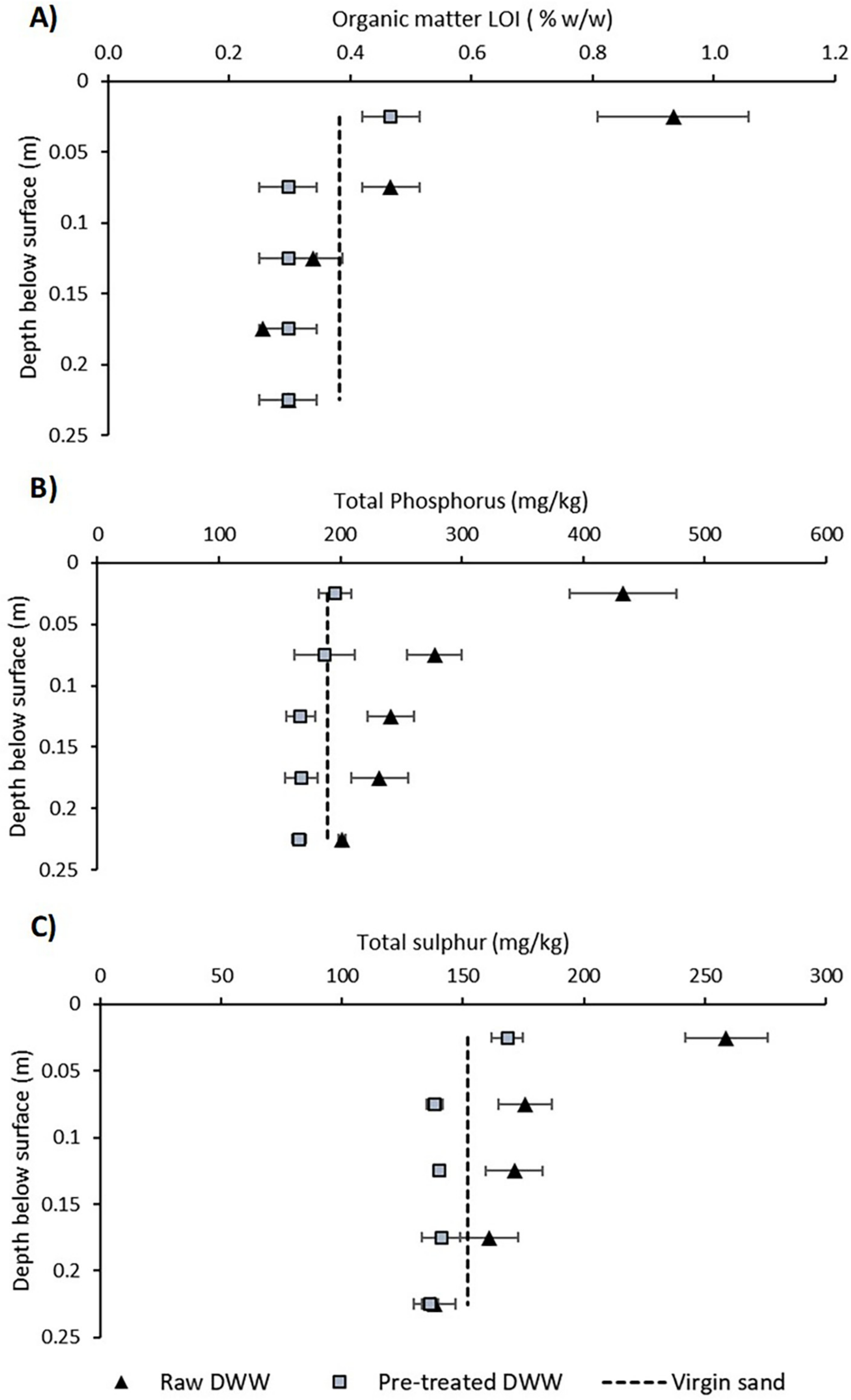

Fig. 5. Chemical properties (mean ± SD) of the filters measured at depths below the sand filter surface at the end of the study: A) organic matter (OM); B) total phosphorus (TP); and C) total sulphur (TS).

Statistical analysis showed that the depth had significant effect ($p < 0.001$) on the chemical properties for both raw and pre-treated DWW filters. However, this difference was most significant between the uppermost layer and all lower layers ($p < 0.001$; Table S2), but was not significant between the lower layers ($p > 0.05$; Table S2), supporting the finding that clogging is a surface phenomenon. The bacterial biomass in raw DWW filters was uniform and homogeneous because the filter was fully utilized when approaching the clogging point, while bacterial biomass in pre-treated DWW filters remained non-uniform as many spatial spots in the filter remained intact (e.g., with less biomass, or without biomass).

3.5. Factors influencing filter clogging and cost of clogging mitigation

In general, OLR, TSSLR, HLR and DF are all important factors that should be considered in designing ISFs. The OLR and TSSLR should not exceed 35 g COD m^2 d^{-1} and 15 g TSS m^2 d^{-1}, respectively (Healy et al.,

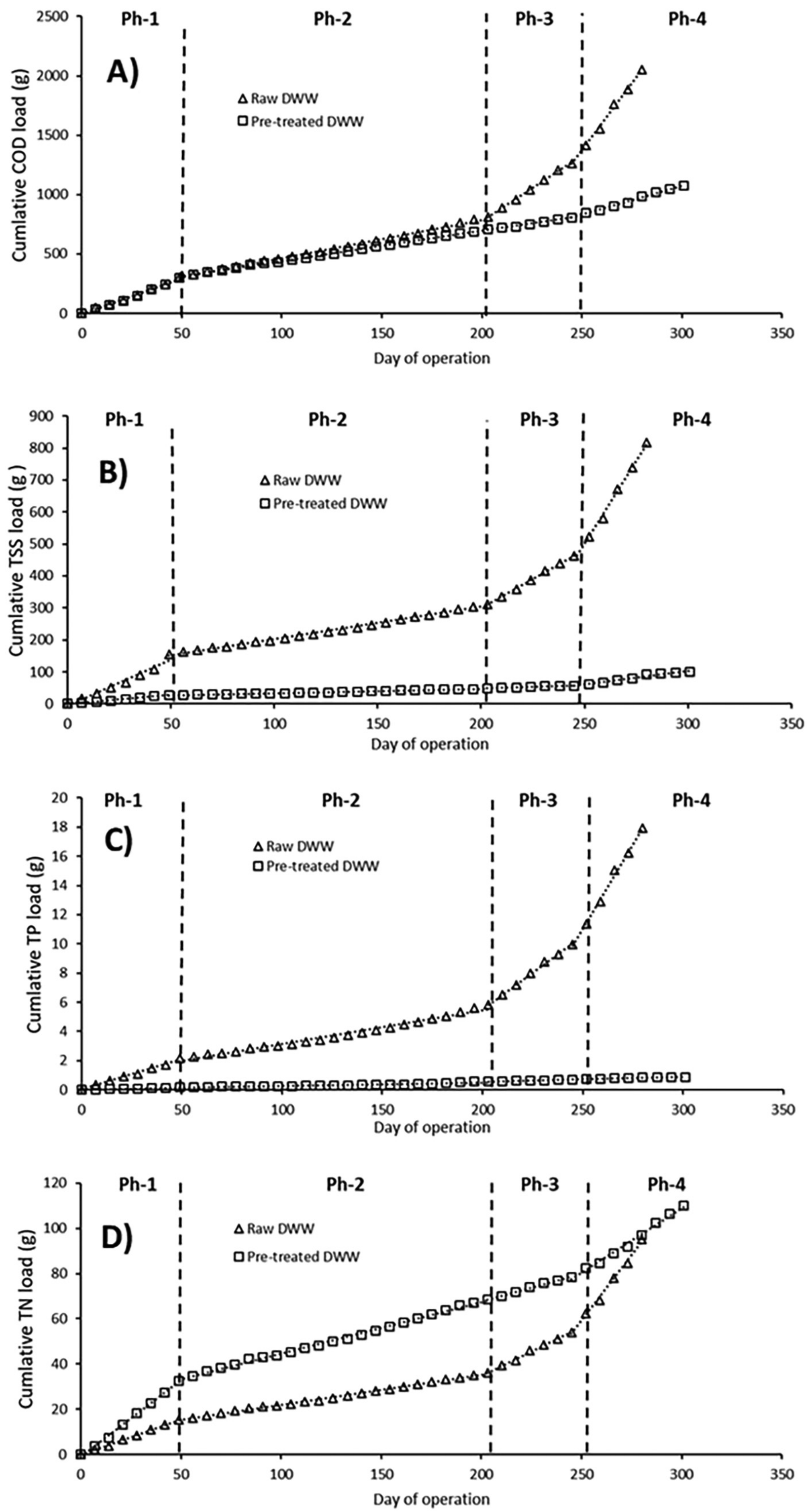

Fig. 6. Cumulative loads of pollutants on raw and pre-treated DWW filters for a study duration of 301 days: A) chemical oxygen demand (COD); B) total suspended solids (TSS); C) total phosphorus; and D) total nitrogen.

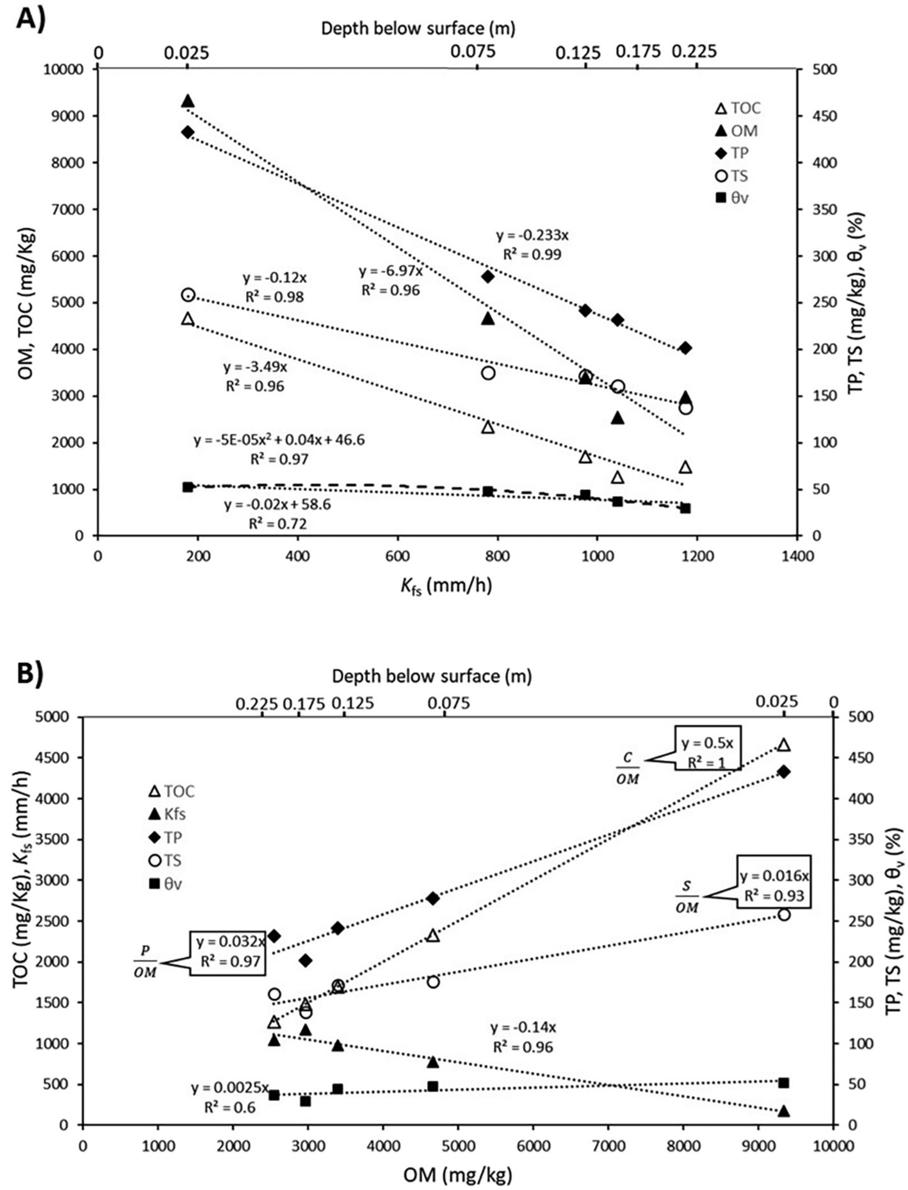

Fig. 7. Inter-relationship between clogging indicators for raw DWW filters: A) against saturated hydraulic conductivity (K_{fs}); B) against organic matter content (OM).

2007; Rodgers et al., 2005; USEPA, 1980). Pre-treated DWW filters complied with these threshold values across the phases, while raw DWW filters exceeded these values in many occasions, especially when operated at the same HLR of pre-treated DWW filters in Phase 3 and 4 (Table 1). Increasing the dosing frequency from 4 to 8 times per day, and doubling the HLR in Phase 4 (Table 1), may have accelerated the clogging of the raw DWW filters. Leverenz et al. (2009) suggested that ISFs operated at high dosing frequencies, for a certain influent COD concentration, encourage continuous heterotrophic biomass development at the surface, which is associated with early clogging, while low dosing frequencies were found to result in stable growth conditions, and therefore long-term steady operation. The low dosing frequencies extends the resting period between doses, which allows for biomass endogenous decay and recovery of the filter porosity (Leverenz et al., 2009). Chen et al. (2021b) point out that the lifetime of an ISF is adversely related to wastewater strength and HLR, and suggested that increasing the HLR can lead to more biological removal burdens to layers underneath the clogging development zone, and therefore negatively impact the operational lifetime of the filters.

The decision to select the coagulation treatment as a pre-treatment step depends on the cost of the chemical coagulant versus the cost of scraping and replacing the surface of sand layer for conventional systems. For example, under operating conditions such as slow filtration of wastewater and water supply treatment systems, scraping the surface would not be cheaper than the use of the coagulant pre-treatment (Table 2). In addition, the land requirement and construction cost of hybrid coagulation-ISFs will be 75 % less than conventional ISF system due to the high reduction in OM/COD achieved by coagulants in this study (Table 2).

4. Conclusion

This study found that pre-treatment of wastewater increased the longevity of ISFs and prevented clogging of the filter media, allowing for longer operation period than conventional ISFs (without a pre-treatment step). During operation, filters receiving raw DWW exhibited higher moisture content than filters receiving pre-treated DWW, indicating that the development of biomass and accumulation of particulate matter was faster in raw

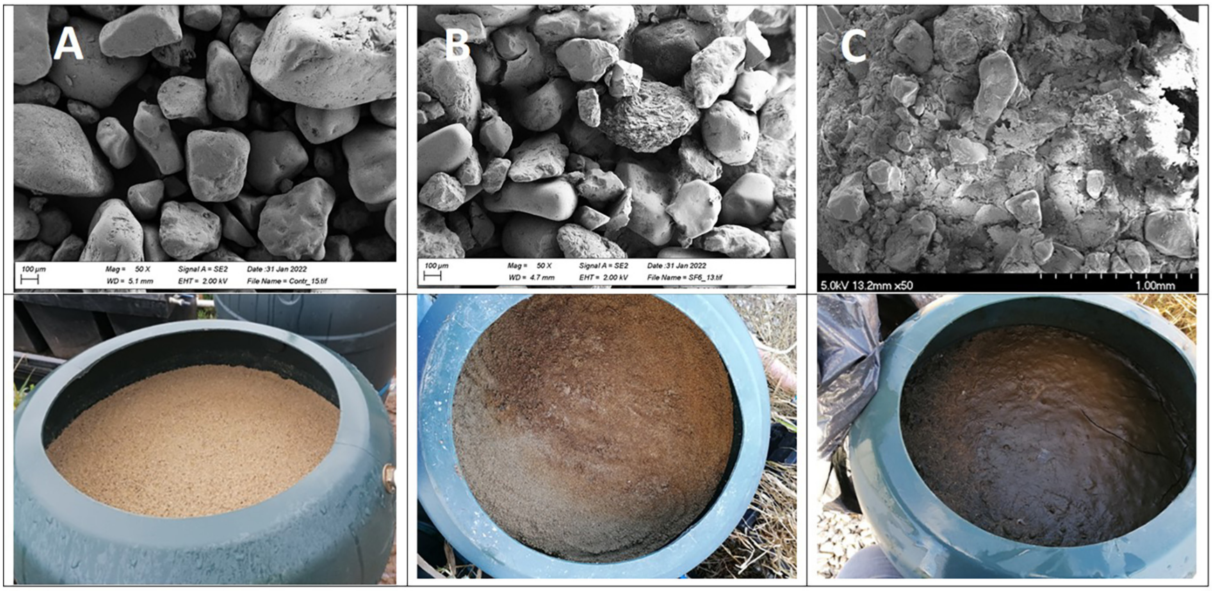

Fig. 8. Scanning electron microscopy (magnification 50X; top row) and camera photography (bottom row) of the surface of the ISFs, where: A) virgin sand sample; B) pre-treated DWW filters and C) raw DWW filters.

DWW filters. Filters receiving raw DWW clogged on day 280, while filters receiving pre-treated DWW had no clogging over the study duration. The $FeCl_3$ prevented clogging and contributed to 45 % of ISF clogging alleviation. Build-up of OM and suspended solids on the surface of raw DWW ISFs appeared to be the main mechanisms responsible of clogging on these filters. In all cases, the clogging indicators reduced with depth from the sand surface and returned to its virgin sand values at deeper depths (i.e. 0.2–0.25 m). Overall, filters without pre-treatment steps are likely to clog faster than filters treating pre-treated wastewater; therefore requiring large surface area for treatment and extensive maintenance.

CRediT authorship contribution statement

Ahmed Mohamed Methodology, Formal analysis, Investigation, Data Curation, Writing (Original Draft), Writing (Reviewing and Editing), Visualisation. **Pat Tuohy** Conceptualization, Methodology, Resources, Writing (Reviewing and Editing), Supervision, Project Administration, Funding Acquisition. **Mark G. Healy** Conceptualization, Methodology, Resources, Writing (Reviewing and Editing), Supervision. **Daire Ó hUallacháin** Methodology, Formal analysis, Writing (Reviewing and Editing), Supervision. **Owen Fenton** Methodology, Writing (Reviewing and Editing), Supervision. **Alma Siggins** Methodology, Writing (Reviewing and Editing), Supervision.

Data availability

Data will be made available on request.

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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Table 2
Comparisons and breakdown of costs[a] for conventional ISF (without coagulation pre-treatment) and hybrid coagulation-ISF system.

	Area requirement[b]	Land cost[c]	Qty of sand[d]	Sand cost[e]	Frequency of scraping[f]	Cost of sand replacement[g]	Qty of $FeCl_3$[h]	Cost of chemical coagulant[i]	Capital costs[j]	Operational costs[k]
	m^2	€	m^3	€	times yr^{-1}	€ yr^{-1}	m^3	€ yr^{-1}	€	€ yr^{-1}
Conventional ISF	600	1645	390	31,200	1	4800	0	0	32,845	4800
Hybrid coagulation-ISF	145	400	94	7520	0.5	578	2	1040	7920	1618

[a] Calculations based on a typical Irish dairy farm with 100 cows.
[b] Surface area was calculated based on an OLR of 30 g m^{-2} d^{-1} for the ISFs and wastewater production of 33 L $cow^{-1}d^{-1}$ (Minogue et al., 2015).
[c] Land cost was calculated based on 10,962 € $acre^{-1}$ (SCSI/Teagasc report, 2022).
[d] Volume of sand was calculated based on sand depth of 0.65 m.
[e] Sand cost was calculated based on commercial sand available at the market (50 € ton^{-1}) and sand density of 1.6 ton m^{-3}.
[f] Estimated based on the current study.
[g] Estimated based on replacement of the top 10 cm of sand layer.
[h] Quantity of $FeCl_3$ was calculated based on dosage rate of 2 mL $FeCl_3$ L^{-1} of DWW, and based on 10,000 L of DWW is produced per cow per year (Minogue et al., 2015).
[i] Cost was estimated using the prices of commercial $FeCl^3$. (520 € m^{-3}).
[j] Capital costs consists of land and sand costs.
[k] Operational costs consist of chemical coagulant and regular cost of replacing sand due to the clogging.

(Teagasc Johnstown Castle), and Emma McDermott (Centre for Microscopy and Imaging, University of Galway).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2023.162605.

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