



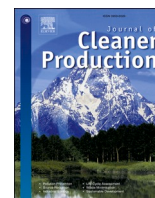
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A novel hybrid coagulation-intermittent sand filter for the treatment of dairy wastewater

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ABSTRACT

An intermittent sand filter (ISF) is a simple and cost-effective treatment method that may be adopted on farms to treat dairy wastewater (DWW). However, the use of ISFs has been limited due to the large area required for treatment, and the risk of filter clogging and phosphorus (P) breakthrough, which decrease the operational lifetime. To overcome these limitations, this study uses a novel, pilot-scale coagulation-sedimentation process prior to loading ISFs with DWW. The performance and operational lifetime of this new hybrid coagulation-ISF system was compared to a conventional ISF system in a replicated outdoor pilot-scale experiment over a 43-wk study duration (covering an entire milking season on a farm in Ireland). The hybrid system was able to operate effectively at a higher hydraulic loading rate than a conventional ISF system. The effluent quality from the conventional ISF deteriorated over the timeframe of the study until clogging occurred, while the hybrid system continued to perform effectively without any evidence of clogging or P breakthrough. The hybrid system obtained removal efficiencies $\geq 99\%$ for all measured water quality parameters (chemical oxygen demand, total suspended solids, total P, ammonium and turbidity), and complied with EU directives concerning urban wastewater treatment. Overall, the hybrid coagulation-ISF is a promising technology that requires a small area (75% reduction in footprint in comparison to a conventional ISF) and minimal operator input, and produces high effluent quality.

1. Introduction

Feeding the world's population in a sustainable manner is one of the key challenges facing agriculture. The recent (2015) removal of the milk quota restrictions has resulted in an expansionary phase in dairy farming in a number of European Union (EU) countries (Micha et al., 2017; DAFM, 2015). This expansion has resulted in an increase in the size of dairy herds (Kelly et al., 2020) and an associated increase in the volumes of dairy wastewater (DWW) generated by farms.

Dairy wastewater (also referred to as dairy soiled water, farm dairy effluent, and dairy dirty water) is effluent from milking parlours, collecting yards, roadways, and other hard-standing areas, and consists of a dilute mixture of cow faeces, urine, milk, detergents and sediment. Land application is the primary disposal method for DWW (Wang et al., 2004; Martínez-Suller et al., 2010). However, when applied at rates that exceed the nutrient demand of the herbage, or when applied under unfavourable soil and weather conditions, land application of DWW can

result in pollutant loss to nearby receiving water bodies, leading to excessive algal growth (eutrophication) and deterioration of the aquatic ecosystem (Sommer and Knudsen, 2021). Hence, to support the objective of attaining good status of surface and ground waters, there is a need for low energy, cost-effective, and low maintenance on-farm treatment processes for DWW that result in an improved effluent quality and reduced risk of pollution.

Intermittent sand filters (ISFs) are simple, cost-efficient, and effective at removing contaminants from DWW (Rodgers et al., 2005; Healy et al., 2007). Intermittent dosing facilitates media aeration between doses, which encourages bacterial growth by aerobic metabolism of organic matter, and hence sufficient aerobic biological wastewater treatment can be achieved with significant reduction of chemical and biochemical oxygen demand (COD and BOD), total suspended solids (TSS), coliform bacteria and viruses (Murnane et al., 2016). Nevertheless, the application of ISFs is limited by the large surface area required for treatment, as a low organic loading rate (OLR) of less than 22 g biochemical oxygen

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demand (BOD) $\text{m}^{-2} \text{d}^{-1}$ is recommended (US Environmental Protection Agency, 1980). Furthermore, regular clogging of ISFs due to accumulation of suspended solids and the development of a microbial biofilm on the filter surface is a major limitation for ISF operation (de Matos et al., 2018). ISFs also have poor long-term phosphorus (P) removal due to the limited capacity of the sand to adsorb P (Torrens et al., 2009; Murnane et al., 2016).

Pre-treatment for ISFs is recommended (US Environmental Protection Agency, 1980; Healy et al., 2007) to reduce the strength of the influent wastewater, and therefore overcome their inherent operational problems, such as the likelihood of filter clogging. Selection of the pre-treatment method depends on the treatment efficiency and resources available on farms e.g. land availability, initial and operation costs, and ease of operation and maintenance. For example, an ISF in combination with a retention pond (Torrens et al., 2009; Chen et al., 2021b) may be a solution that minimises energy and cost. However, the application of a retention pond is limited due to their large surface area and their poor ability to remove nutrients (Fyfe et al., 2016). To overcome some of these limitations, a coagulation pre-treatment method was used in the current study as it is efficient, requires a small treatment area, and is cost-effective when small doses are utilised. Furthermore, management of the dosing step is simple and can be operated by the landowner.

Pre-treatment of DWW through a conventional coagulation- sedimentation process may be a holistic approach to overcome the operational problems and shortcomings of ISFs. Cameron and Di (2019) achieved a high reduction of COD, TSS and total phosphorus (TP) for DWW treated with a ferric-based coagulant at low doses. Consequently, this approach can result in a smaller sized downstream ISF, with a potentially longer operational period, without clogging and without P breakthrough. However, there is little to no awareness of their efficiency when combined with ISFs in the treatment of DWW over an entire milking period on a farm. Only one study has investigated a combined coagulation-ISF system, but this was in the treatment of greywater and the study duration was only 60 days (Singh et al., 2021). That study reported stable and reliable performance for the parameters investigated (COD, BOD and faecal coliforms), and produced effluent suitable for reuse.

In this study a combined pilot-scale coagulation-ISF system for DWW treatment was studied for the first time and compared to a conventional ISF system. The performance and lifespan of both systems were compared across four experimental phases, utilising different OLRs and hydraulic loading rates (HLRs), over a 43-week duration (covering a complete milking season). This study is the first to treat actual DWW at

pilot-scale (outdoors) under typical Irish climatic conditions.

2. Materials and methods

2.1. Wastewater collection and sand filter design

Fresh DWW was collected weekly for the duration of the experiment in a 1000 L capacity intermediate bulk container (IBC; Fig. S1–A) from an access chamber on a DWW discharge pipe at the Teagasc Moorepark Dairy farm, Fermoy, Co. Cork, Ireland (52°09'42.0"N 8°15'09.7"W). A submersible pump, placed at the bottom of the access chamber, was used to fill the IBC tank with DWW during milking events (Fig. 1-A). The DWW comprised washings from the milking parlour and collecting yard, and from cleaning the milking plant.

The DWW was allowed to stand overnight in the IBC tank in order to allow large particulate matter to settle to the bottom of the IBC tank, and therefore prevent system blockage, and maintain the integrity of the downstream pumps over the study duration. Following this, 200 L of DWW was drained from the upper part of the IBC tank and fed by gravity into a 210 L cylindrical plastic tank (hereafter referred to as raw DWW; Fig. 1-A). The raw DWW was agitated regularly to ensure homogeneity.

The remaining DWW in the IBC tank (800 L) was mixed with 2 L of a chemical flocculant ferric chloride solution (FeCl_3 ; 40% w/w; Table S1) (440 mg Fe L^{-1} of DWW; $10.35 \text{ g Fe g}^{-1} \text{ P}$) at a velocity gradient of 900 s^{-1} for 10 min, using an IBC mixer mounted on the IBC tank. Mohamed et al. (2020) showed that this concentration and mixing time were optimal for the removal of pollutants found in DWW. The ferric salt coagulants are usually effective at low pH (Bratby, 2016; Naceradska et al., 2019), therefore the FeCl_3 solution had a pH of 1–2 (Table S1). Following mixing, the mixture was allowed to settle for 3 h, after which 200 L of the supernatant was drained from the upper part of the IBC tank and fed by gravity into another separate 210 L cylindrical plastic tank (hereafter referred to as supernatant; Fig. 1-B). This provided sufficient wastewater to supply the ISFs for one week. The IBC, raw DWW, supernatant and collection tanks were fully emptied and thoroughly cleaned every week before the preparation of the new weekly batches of DWW.

Raw DWW and supernatant characteristics are presented in Table 1, and were pumped intermittently 4 or 8 times per day (Table 2) on to replicated ($n = 3$) single-layer sand filters, using diaphragm waste pumps controlled by electronic timers (Fig. 1-B). The wastewater was distributed over the filter surface using uPVC distribution manifolds. Depending on the experimental phase, hydraulic or organic loading rates were adjusted using manual flow control valves that were located

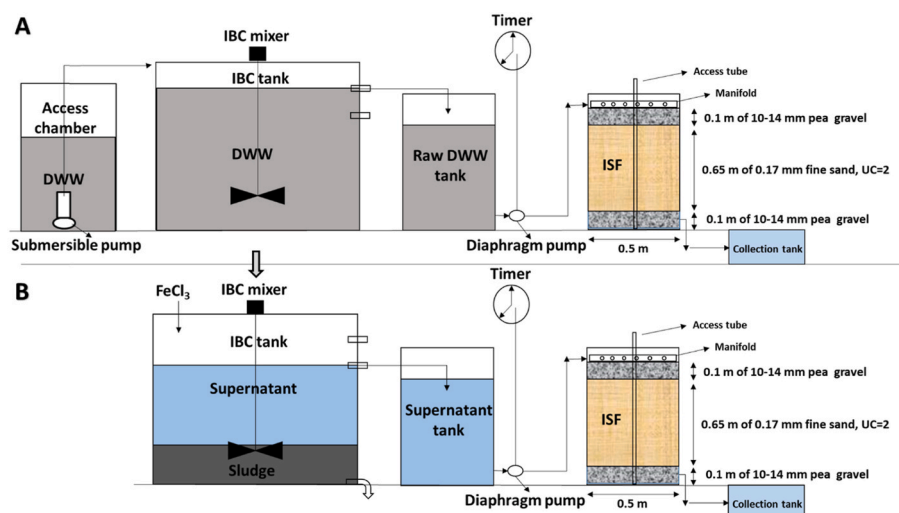


Fig. 1. Experimental set-up: A) Schematic view of raw DWW system; B) Schematic view of supernatant system.

Table 1

Influent raw DWW and supernatant characterization: mean \pm standard deviations (SD; n = 34).

Parameter	Raw DWW	Supernatant	Pre-treatment efficiency (%)
COD (mg L ⁻¹)	5385 \pm 1212	1311 \pm 375	75.7 (***)
Turbidity (NTU)	1976 \pm 610	85 \pm 80	95.7 (***)
TSS (mg L ⁻¹)	1975 \pm 825	108 \pm 96	94.6 (***)
TP (mg L ⁻¹)	42.5 \pm 15.0	1.14 \pm 1.0	97.3 (***)
DRP (mg L ⁻¹)	19.3 \pm 12.04	0.21 \pm 0.51	98.9 (***)
TN-N (mg L ⁻¹)	240.5 \pm 60.3	129.6 \pm 40.3	46.1 (***)
TNp-N (mg L ⁻¹)	75.6 \pm 31	4.3 \pm 7.2	94.3 (***)
NH ₄ -N (mg L ⁻¹)	118.4 \pm 40.8	87 \pm 30.4	26.5 (***)
DON-N (mg L ⁻¹)	49.2 \pm 25.3	34.8 \pm 23	29.2 (*)
TON-N (mg L ⁻¹)	0.8 \pm 2.1	3.9 \pm 4.4	N/A
pH	7.35 \pm 0.57	6.1 \pm 0.9	17.3 (***)
Cl (mg L ⁻¹)	155 \pm 66	833 \pm 145	N/A

Statistically significant differences between Raw DWW and supernatant are shown at $P < 0.001$ as ***; $P < 0.01$ as **; $P < 0.05$ as * and no significant difference as NS.

Table 2

Experimental phases of different operational regimes of OLR and HLR applied to raw DWW and supernatant filters during a period of 43 weeks.

Operation mode	Phase	Weeks	Waste source	OLR (g m ⁻² d ⁻¹) Mean \pm SD	HLR (L m ⁻² d ⁻¹) Mean \pm SD	Dosing frequency
Same OLR	1	1 to 7	Raw DWW	30 \pm 6	6 \pm 1.5	4
			Supernatant	30 \pm 6	20 \pm 4	
	2	8 to 29	Raw DWW	15 \pm 5	3.0 \pm 0.75	4
			Supernatant	15 \pm 5	10 \pm 2	
Same HLR	3	30 to 35	Raw DWW	55 \pm 8	10 \pm 2	4
			Supernatant	15 \pm 5	10 \pm 2	
	4	36 to 43	Raw DWW	110 \pm 10	20 \pm 4	8
			Supernatant	30 \pm 6	20 \pm 4	

downstream of the pumps, and/or by changing the pumping duration (typically less than 1 min per dose). All filters were operated in single-pass mode for the entire experimental period. The treated/filtered water exited each filter through a 0.04 m outlet pipe to a 20 L capacity collection tank.

Unlike previous studies, in which sand filters were used to treat DWW at laboratory-scale (Rodgers et al., 2005; Ruane et al., 2014; Murnane et al., 2016), all sand filters in the current study were located outdoors and constructed using 210 L capacity plastic barrels (reinforced in the middle to prevent expansion: Fig. S1–C). Each barrel had an internal diameter of 0.5 m and a total depth of 0.9 m, including 0.05 m of free-board. The design specifications were based on US EPA guidelines (US Environmental Protection Agency, 1980). A 0.1 m layer of clean pea gravel with a particle size of 10–14 mm was placed at the base of each barrel to prevent washout of the filter media. Each barrel was then filled with fine sand (effective size, $d_{10} = 0.17$ mm; uniformity coefficient, UC = 2) to a depth of 0.65 m by adding it in eight lightly compacted increments. The top layer of the filter was covered by a 0.1 m deep layer of washed pea gravel, with a particle size of 10–14 mm. The filters were partially covered to prevent rainfall ingress, while allowing air to circulate in the filter. In 2021, the mean annual temperature at the study site was 10.5 °C. The approximate mean seasonal temperatures for the region were as follows: winter, 5.4 °C; spring, 8.2 °C; summer, 15.7 °C; and autumn, 11.7 °C (Met Éireann, 2021). The total annual rainfall and potential evaporation were 1013 and 716 mm, respectively (Met Éireann, 2021).

2.2. Start-up operation and experimental phases

Prior to operation, 500 L of potable water was pumped onto each filter over a period of five days to clean any organic material from the media. On day 15 of operation, in order to enrich the filters with bacteria, and accelerate microbial growth, each filter was seeded with 4 L of nitrifying activated sludge (mixed liquor suspended solids, MLSS = 4350 mg L⁻¹; sludge volume index, SVI = 124) collected from a local wastewater treatment plant, which was used to treat a mixture of domestic and dairy industry wastewater.

The study spanned a complete milking season (February to December; 43 weeks). Spring-calving dairy herds are not milked for 8–10 weeks pre-calving, so there is no DWW produced during this period at the study site (typically early December to mid-February). The experiment consisted of four phases (Table 2). All ISFs were operated with the same OLR in the first and second phases. In Phase 1 (Wk 1–Wk 7), the operational OLR was set at upper design limits (30 \pm 6 g COD m⁻² d⁻¹) (after Rodgers et al., 2005). However, to prevent clogging of the filters, it was decided to reduce the OLR in Phase 2 to 15 \pm 5 g COD m⁻² d⁻¹ for both sets of filters (Wk 8 to Wk 29). Hydraulic loading rates were adjusted during Phases 1 and 2 to accommodate the varying organic loads. In the third and fourth phases, all filters were operated with the same HLR. In Phase 3 (Wk 30 to Wk 35), the ISFs receiving supernatant continued to be operated with the same conditions as in Phase 2 (OLR = 15 \pm 5 g COD m⁻² d⁻¹, HLR = 10 L m⁻² d⁻¹). The HLR in the raw DWW ISFs was increased to 10 L m⁻² d⁻¹, producing an OLR of 55 \pm 8 g COD m⁻² d⁻¹. In Phase 4, the HLR was further increased to 20 L m⁻² d⁻¹ for both sets of filters, to examine the efficacy of the raw DWW and supernatant ISFs at elevated OLRs. The rationale behind Phase 3 and 4 was to prove that pre-treatment has a significant positive effect on the performance of the hybrid system, and that conventional ISFs can fail if they are operated at the same HLR as a hybrid system due to the high OLR.

2.3. Analysis

Samples were collected weekly for analysis from the influent raw DWW tank (n = 1; bulk sample) and supernatant tank (n = 1; bulk sample), and from the effluent collection tanks (n = 3 for both the raw DWW and supernatant ISFs). The samples were preserved at -20 °C (for COD) or 4 °C (for all other parameters) and analysed within 14 days. Temperature and pH were measured immediately using an HQ40d Multi Meter (HACH, USA), and turbidity was measured using a portable turbidity meter (Orion AQUAfast AQ3010, ThermoFisher Scientific, USA) and expressed as Nephelometric Turbidity Units (NTU). COD was measured using the dichromate method. Total suspended solids were measured by filtering a 100 mL sample through a Whatman GF/C (pore size 1.2 μ m) filter paper using a vacuum pump, and drying the filter paper for 2 h at 103–105 °C. Total nitrogen (TN), filtered TN (TN_F), TP, and filtered TP (TP_F) were measured using the Persulphate Oxidative Digestion method. Dissolved reactive phosphorus (DRP), ammonium (NH₄-N), nitrite (NO₂-N), total oxidised N (TON) and chloride (Cl) were analysed spectrophotometrically, following filtration through 0.45 μ m filters, using a nutrient analyser (Aquakem 600A/Konelab 60, Thermo Clinical Labsystems, Vantaa, Finland). Nitrate (NO₃-N) was calculated by subtracting NO₂-N from TON. Dissolved organic N (DON) was calculated by subtracting TON and NH₄-N from TN_F. Particulate N (TN_P) was calculated by subtracting TN_F from TN. Dissolved organic P (DOP) was calculated by subtracting DRP from TP_F. Particulate phosphorus (PP) was calculated by subtracting TP_F from TP. All tests were carried out in accordance with the standard methods (APHA, 2005).

The sand filters were instrumented with 1 m-deep access tubes (Fig. 1; type ATL1, Delta-T Devices Ltd., Cambridge, UK) to allow the volumetric moisture content to be determined at different depths. As biofilm is hydrophilic, its build-up in a filter may be measured, by proxy, by time-domain reflectometry (TDR) (Rodgers et al., 2004). In order to

monitor the biomass build-up (and the potential risk of filter clogging) after steady-state was achieved (from Wk 11 onwards), the volumetric water content (θ_v) was recorded weekly for the top 25 cm of the sand layer and measured incrementally every 0.05 m (5 layers) during the last hour of each dosing cycle. A TDR profile probe (type PR1/6d-02, Delta-T Devices Ltd., Cambridge, UK) was inserted in the access tubes, and 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, all ISFs were deconstructed, and the biomass build-up on filters was characterised in 0.05 m increments to a total depth of 0.25 m below the sand surface. Total organic carbon (TOC) was used as an indication of biomass distribution within the filter, and was measured using the DUMAS combustion method (BS EN 15936, BSI, 2012).

2.4. Data analysis

Statistical analyses were carried out using SAS 9.4 (SAS Institute Inc., USA). Repeated measure modelling was undertaken using PROC MIXED. PROC MIXED facilitated the unbalanced replication associated with the data and addressed challenges associated with non-normal distribution. The model included the following factors: Treatment (four treatments: influent raw DWW, influent supernatant, effluent from raw DWW ISF, effluent from supernatant ISF), Week (multiple weeks that varied from phase to phase), and the interaction between these factors (Treatment x Week) as fixed terms. The specific ISFs were treated as a repeated measure. LSMEANS statement (with a Tukey adjustment) identified where significant differences occurred between treatments. Four separate models were constructed, a separate model for each phase of the experiment, to account for methodological differences between phases as described in Section 2.2. Probability values of $P > 0.05$ were deemed not significant.

3. Results and discussion

3.1. Organic matter removal

The use of $FeCl_3$ flocculant for the pre-treatment of DWW reduced COD significantly ($P < 0.001$; representing a reduction of 76%; Table 1). This result was comparable with Mohamed et al. (2020), who achieved 84% removal with an effluent concentration of 1600 mg L^{-1} for DWW treated with $FeCl_3$ at the same dosage (470 mg Fe L^{-1} ; $10.83 \text{ g Fe g}^{-1} \text{ P}$).

In Phase 1, to obtain a consistent OLR of $30 \text{ g COD m}^{-2} \text{ d}^{-1}$ for all filters, the HLR of the ISFs treating supernatant ($20 \text{ L m}^{-2} \text{ d}^{-1}$) was 3.3 times higher than that of ISFs treating raw DWW ($6 \text{ L m}^{-2} \text{ d}^{-1}$). This was due to the fact that the supernatant had a lower COD concentration than raw DWW, following $FeCl_3$ amendment. In Phase 1, the effluent concentration of COD from the ISFs treating raw DWW was consistently lower ($P < 0.001$) than from the ISFs treating supernatant (Fig. 2), although steady-state operation was not achieved during this phase.

In Phase 2, the OLR was reduced by half ($15 \text{ g m}^{-2} \text{ d}^{-1}$) to prevent clogging, by adjusting the HLR to 3 and $10 \text{ L m}^{-2} \text{ d}^{-1}$ for raw DWW and supernatant ISFs, respectively. The systems reached steady-state/stable operation conditions during this phase, when the ISFs were fully biologically active and consistent COD, TSS, N and P effluent concentrations were achieved. During the steady-state operation of Phase 2, there was no differences in the effluent COD between raw DWW and supernatant ISFs ($P > 0.05$; Fig. 2), and both effluents concentrations were far below the limit values for discharge to urban waters (125 mg L^{-1} ; 91/271/EEC; EEC, 1991). Mohamed et al. (2020) suggested that the reduction in COD by $FeCl_3$ was due to the removal of particulate COD, and that the remaining COD in the supernatant was likely in soluble form. This would suggest that soluble COD in raw DWW accounted for about 25% of the total influent COD (Table 1), with much of the influent COD being associated with the particulate fraction. Therefore, it was

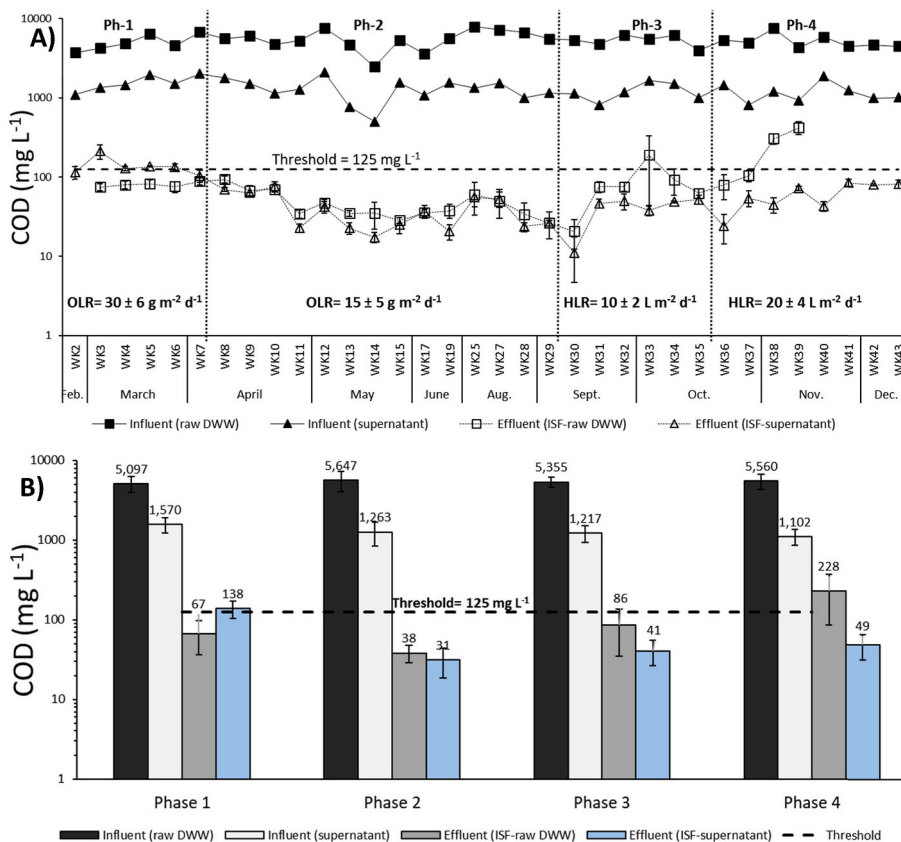


Fig. 2. Influent and effluent chemical oxygen demand (COD) concentrations of raw DWW and supernatant filters for a study period of 43 weeks: A) on a weekly basis; B) per phase.

likely that physical filtration was the primary removal mechanism for COD in the case of raw DWW ISFs. On the other hand, the aerobic nature of the ISFs would suggest that oxidation of organic matter was the primary mechanism contributing to the decrease in COD in supernatant ISFs.

In Phase 3, when the operational HLR of the raw DWW ISFs was increased to match that of the supernatant ISFs ($10 \text{ L m}^{-2} \text{ d}^{-1}$), the OLR increased to $55 \text{ g COD m}^{-2} \text{ d}^{-1}$ for the raw DWW ISFs. The EPA guidelines (US Environmental Protection Agency, 1980) recommend that the OLR should not exceed $22 \text{ g BOD m}^{-2} \text{ d}^{-1}$ (equivalent to $35 \text{ g COD m}^{-2} \text{ d}^{-1}$). Despite this increase in OLR, the raw DWW ISFs showed a good degree of robustness and produced an effluent COD below the EU directive standard (125 mg L^{-1} ; Fig. 2-A). However, supernatant ISFs, with the lower OLR of $15 \text{ g COD m}^{-2} \text{ d}^{-1}$, performed significantly better ($P < 0.05$; Fig. 2).

In Phase 4, when the operational HLR was increased to $20 \text{ L m}^{-2} \text{ d}^{-1}$ for both set of ISFs, the OLR increased to $110 \text{ g COD m}^{-2} \text{ d}^{-1}$ for the raw DWW ISFs and $30 \text{ g COD m}^{-2} \text{ d}^{-1}$ for the supernatant ISFs. As a result of the high OLR applied to the raw DWW ISFs, their ability to remove COD deteriorated, with effluent concentrations higher than the supernatant ISF effluent concentration ($P < 0.05$; Fig. 2), and exceeding the threshold value of $125 \text{ mg COD L}^{-1}$. In addition, all raw DWW ISFs exhibited clogging issues during this phase (Wk 40).

3.2. Suspended solids and turbidity removal

The use of FeCl_3 flocculant for the pre-treatment of DWW reduced TSS and turbidity significantly, primarily through sedimentation process ($P < 0.001$; representing a reduction of 95%; Table 1). The results were similar to those reported by Cameron and Di (2019) and Mohamed et al. (2020), which used poly-ferric sulphate and FeCl_3 coagulants to treat DWW at optimum dosages of 214 mg Fe L^{-1} ($6.1 \text{ g Fe g}^{-1} \text{ P}$) and 470 mg

Fe L^{-1} ($10.83 \text{ g Fe g}^{-1} \text{ P}$), respectively.

Physical filtration/screening was the primary removal mechanism of TSS and turbidity in ISFs. During the steady-state operation of Phase 2, both raw DWW and supernatant ISFs were effective at removing TSS to below the standard limit of 35 mg L^{-1} specified by the EU directive for urban water discharge (91/271/EEC; EEC, 1991). However, the supernatant ISFs performed significantly better than raw DWW ISFs ($P < 0.05$; Fig. 3). In this phase, the suspended solids loading rate (SSLR) for raw DWW ISFs ($5.1 \text{ g SS m}^{-2} \text{ d}^{-1}$) was eight times greater than the supernatant ISFs ($0.65 \text{ g SS m}^{-2} \text{ d}^{-1}$).

In Phase 3, the SSLR of raw DWW ISFs was increased to $18 \text{ g SS m}^{-2} \text{ d}^{-1}$ versus $1.1 \text{ g SS m}^{-2} \text{ d}^{-1}$ for supernatant ISFs. Consequently, the mean TSS concentration in the effluent from the raw DWW ISFs exceeded the EU threshold value of 35 mg L^{-1} , and was significantly higher than the supernatant ISFs ($P < 0.001$; Fig. 3). In this phase, the operational SSLR in raw DWW ISFs was higher than the SSLR recommended for successful ISF operation (Healy et al., 2007). For example, Rodgers et al. (2005) and Murnane et al. (2016) operated ISFs with a maximum SSLR of 14.4 and $11 \text{ g SS m}^{-2} \text{ d}^{-1}$, respectively, and achieved acceptable effluent TSS concentrations.

In Phase 4, the SSLR increased to $46 \text{ g SS m}^{-2} \text{ d}^{-1}$ for raw DWW ISFs and 4 for supernatant ISFs. Despite this increase in SSLR for raw DWW ISFs, the mean TSS concentrations reduced in the effluent (compared to Phase 3), but was higher than supernatant ISFs ($P < 0.001$; Fig. 3). This decrease in TSS concentration was likely because the pores of the filters got smaller, as a result of clogging. Ruane et al. (2012) found in laboratory-scale woodchip filters that the removal of TSS improved over time, and suggested that the gradual accumulation of SS in the pore space likely led to more immediate SS removal.

Suspended solids in the wastewater, along with the biofilm generated within the ISF, are the two major factors that cause clogging in ISFs (Healy et al., 2007; de Matos et al., 2018). The pre-treatment step, which

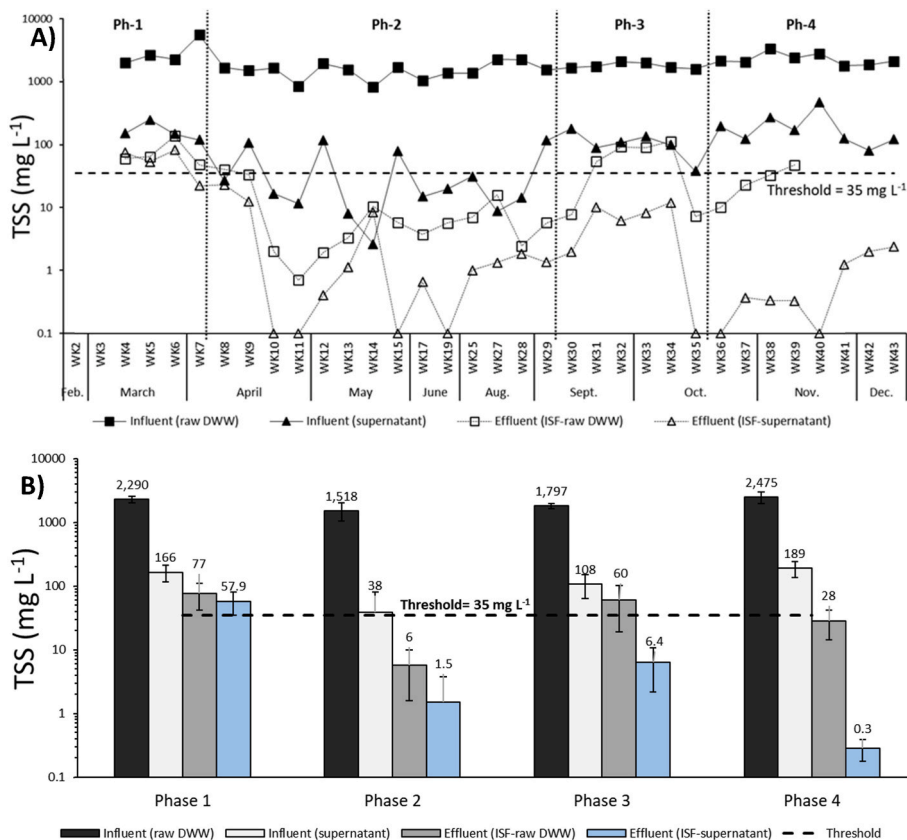


Fig. 3. Influent and effluent total suspended solids (TSS) concentrations of raw DWW and supernatant filters for a study period of 43 weeks: A) on weekly basis; B) per phase.

involves dosing with FeCl₃, significantly reduced the SSLR and OLR in the supernatant ISFs, and therefore prevented the early clogging of supernatant ISFs.

Regarding turbidity, the supernatant ISFs produced very high quality effluent (Fig. 4) that complied with the EU legislation and WHO guidelines for drinking water (turbidity <1 NTU; 98/83/EEC; EEC, 1998; WHO, 2017). Across the phases, the mean effluent turbidity from supernatant ISFs was consistent and significantly lower ($P < 0.001$) than those from raw DWW ISFs (Fig. 4).

3.3. Phosphorus retention

Total P and DRP were reduced significantly by FeCl₃ ($P < 0.001$; representing a reduction of 98%; Table 1). The results of TP and DRP were comparable to those achieved by Fenton et al. (2011), who achieved P effluent concentration less than 1 mg L⁻¹ using FeCl₃ at a stoichiometric rate of 200 g Fe g⁻¹ P for DWW treatment. Chemical precipitation in the form of ferric phosphate (FePO₄) was the main mechanism of P removal by the coagulation-sedimentation process (Bratby, 2016).

During the steady-state of Phase 2, both sets of ISFs were effective at reducing TP to below the standard limit of 1 mg L⁻¹ (91/271/EEC; EEC, 1991, Fig. 5-A). However, supernatant ISFs performed significantly better than raw DWW ISFs ($P < 0.001$; Fig. 5-A). There was no significant difference ($P > 0.05$) in effluent DRP between supernatant and raw DWW ISFs in Phase 2 (Fig. 5-B). Therefore, the difference in TP removal between the two systems was likely due to the raw DWW ISFs' capability to remove the other forms of TP such as DOP and PP. Adsorption and sorption were probably the main mechanisms responsible for DRP and DOP removal, while physical screening was likely the main mechanism responsible for PP removal (Murnane et al., 2016). The TP loading rate on the raw DWW ISFs were higher, by up to two orders of magnitude, than the supernatant ISFs. As a result of this high TP loading rate in raw

DWW ISFs, the TP and DRP concentrations increased linearly in the effluent starting from Wk 30, and exceeded the supernatant influent and the threshold value of 1 mg L⁻¹ at Wk 35, and were significantly higher than effluents from supernatant ISFs ($P < 0.001$; Fig. 5-A, 5-B and 5-C). This indicated that the P adsorption capacity of the filter diminished with time, as all active sorbent sites of sand became exhausted. Phosphorus breakthrough is common in ISFs; for example, Murnane et al. (2016) observed, after 150 days of operation, a rapid DRP breakthrough in ISFs that were used for the treatment of DWW at an OLR of 35 g COD m⁻² d⁻¹. Torrens et al. (2009) also experienced a low P retention after one year of operation and the P removal efficiency dropped drastically from 80% to < 5% for ISFs that were used to treat pond effluent at an OLR ranging from 17 to 170 g COD m⁻² d⁻¹. The cumulative TP mass loaded in the raw DWW ISFs until Wk 40 was approximately 18 g P, and the majority of this load was removed (>95%). Accordingly, the mass of P removed per mass of sand was estimated to be 88 mg kg⁻¹. This value was similar to that measured by Healy et al. (2010), who recorded a maximum adsorption capacity of 85 mg P kg⁻¹ for a sand similar to that used in the current study. Using coagulation as a pre-treatment step offers the possibility to reduce P load significantly and delays P breakthrough.

3.4. Nitrogen conversion

Total nitrogen was also reduced by FeCl₃ ($P < 0.001$), but the removal efficiency was poor compared to removals of COD, turbidity, TSS and TP (Table 1). The main mechanism of N removal by FeCl₃ was due to particulate N removal through sedimentation (94% removal; Table 1). The remaining N in the supernatant mainly comprised NH₄-N and DON-N, which can be only removed through other biological and chemical transformation processes such as nitrification-denitrification, bio-adsorption and volatilization (Henze et al., 2008). Similar to the current study, Cameron and Di (2019) reported a maximum TN removal

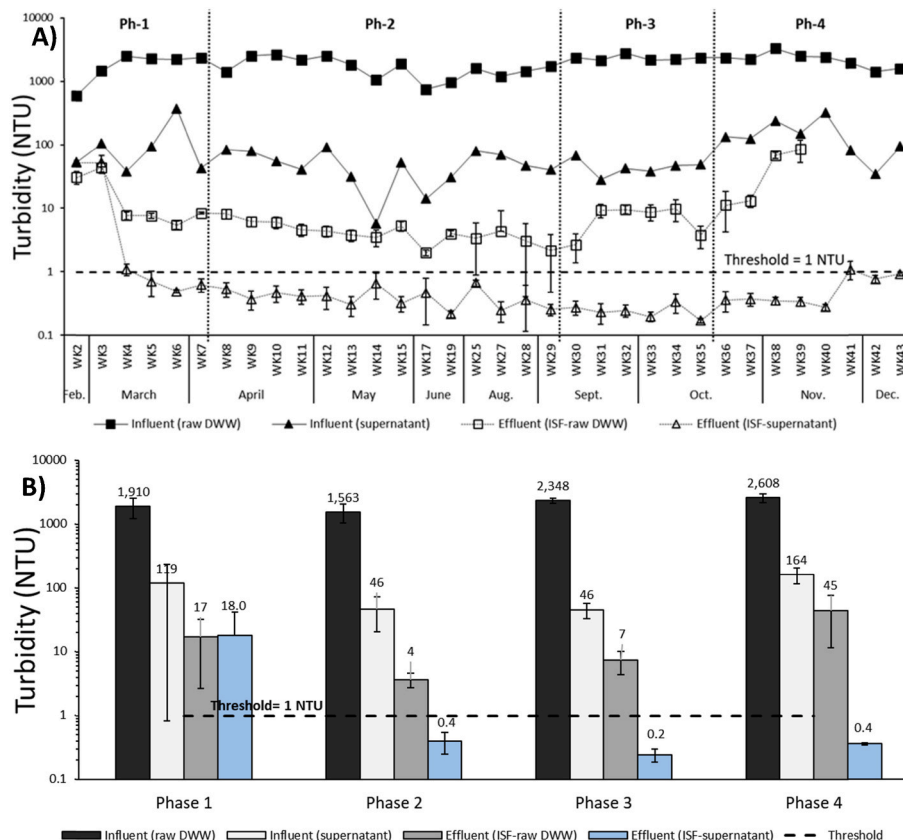


Fig. 4. Influent and effluent turbidity concentrations of raw DWW and supernatant filters for a study period of 43 weeks: A) on weekly basis; B) per phase.

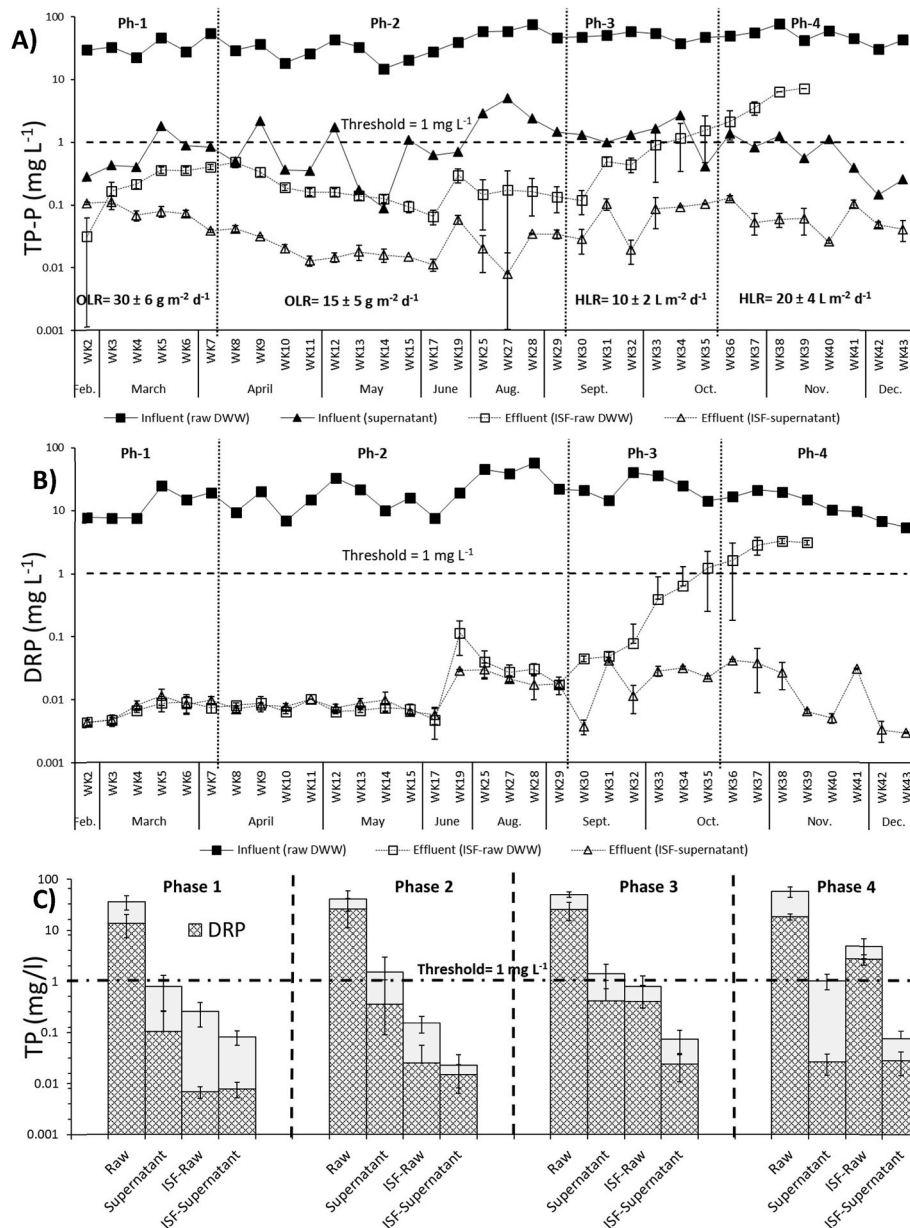


Fig. 5. Influent and effluent concentrations of raw DWW and supernatant filters for a study period of 43 weeks: A) total phosphorus (TP) per week; B) dissolved reactive phosphorus (DRP) per week; and C) total phosphorus and dissolved reactive phosphorus per phase.

of 57%, with a corresponding effluent concentration of 87 mg L^{-1} using poly-ferric sulphate coagulant to treat DWW at an optimum dosage of 214 mg Fe L^{-1} ($6.1 \text{ g Fe g}^{-1} \text{ P}$). The results were also consistent with those achieved by Mohamed et al. (2020), who obtained removals of 35 and 20% for TN and $\text{NH}_4\text{-N}$, respectively, for DWW treated with FeCl_3 at the same dosage.

During the steady-state of Phase 2, the effluent TN concentration from the supernatant ISFs was lower ($P < 0.001$) than raw DWW ISFs (Fig. 6-A; Fig. 7-A). In Phase 3, there was no significant difference ($P > 0.05$) in the effluent TN between raw DWW ISFs and supernatant ISFs (Fig. 6-A; Fig. 7-A). In contrast to Phase 2, the effluent TN from the supernatant ISFs was higher ($P < 0.05$) than raw DWW ISFs (Fig. 6-A; Fig. 7-A) in Phase 4. Across the phases, the effluent TN from both sets of ISFs comprised mainly TON-N (75–95%), followed by DON (5–20%), then Tnp-N (0–6%), and with negligible effluent $\text{NH}_4\text{-N}$ (Fig. 7-B; Fig. S2-B). Physical filtration was the primary removal mechanism for TN in the raw DWW ISFs, as Tnp-N in the influent raw DWW reduced by 95.3% (Fig. 7-A). The supernatant ISFs had capability to remove only

37.3% of the influent supernatant, as the majority of Tnp-N (94.3%; Table 1) was already removed in the upstream process of the coagulation. This make the combined coagulation-ISF system had an overall removal efficiency of 96.6% (Fig. 7-A). Sorption was likely the main mechanism responsible for DON-N removal, and both raw DWW and supernatant ISFs achieved moderate respective removal efficiencies of 62 and 65% (Fig. 7-A). The upstream treatment by FeCl_3 produced 29.2% removal of influent DON-N (Table 1), making the combined system had an overall removal efficiency of 75% for DON-N, which was better than the raw DWW ISFs.

Ammonium in the influent was converted mainly to TON-N in the effluent for both systems. The pH of the effluent raw DWW and supernatant filters was slightly alkaline (Fig. S3), which may have encouraged ammonia volatilization. Nitrification occurred in Phase 1 (Fig. 6-C) in both sets of ISFs, and was fully achieved during Phase 2 (Wk 11; Fig. 6-B). At the steady-state operation of Phase 2, the effluent TON-N from raw DWW ISFs was higher ($P < 0.001$) than supernatant ISFs (Fig. 6-C). This difference was expected as the influent $\text{NH}_4\text{-N}$ for raw DWW ISFs

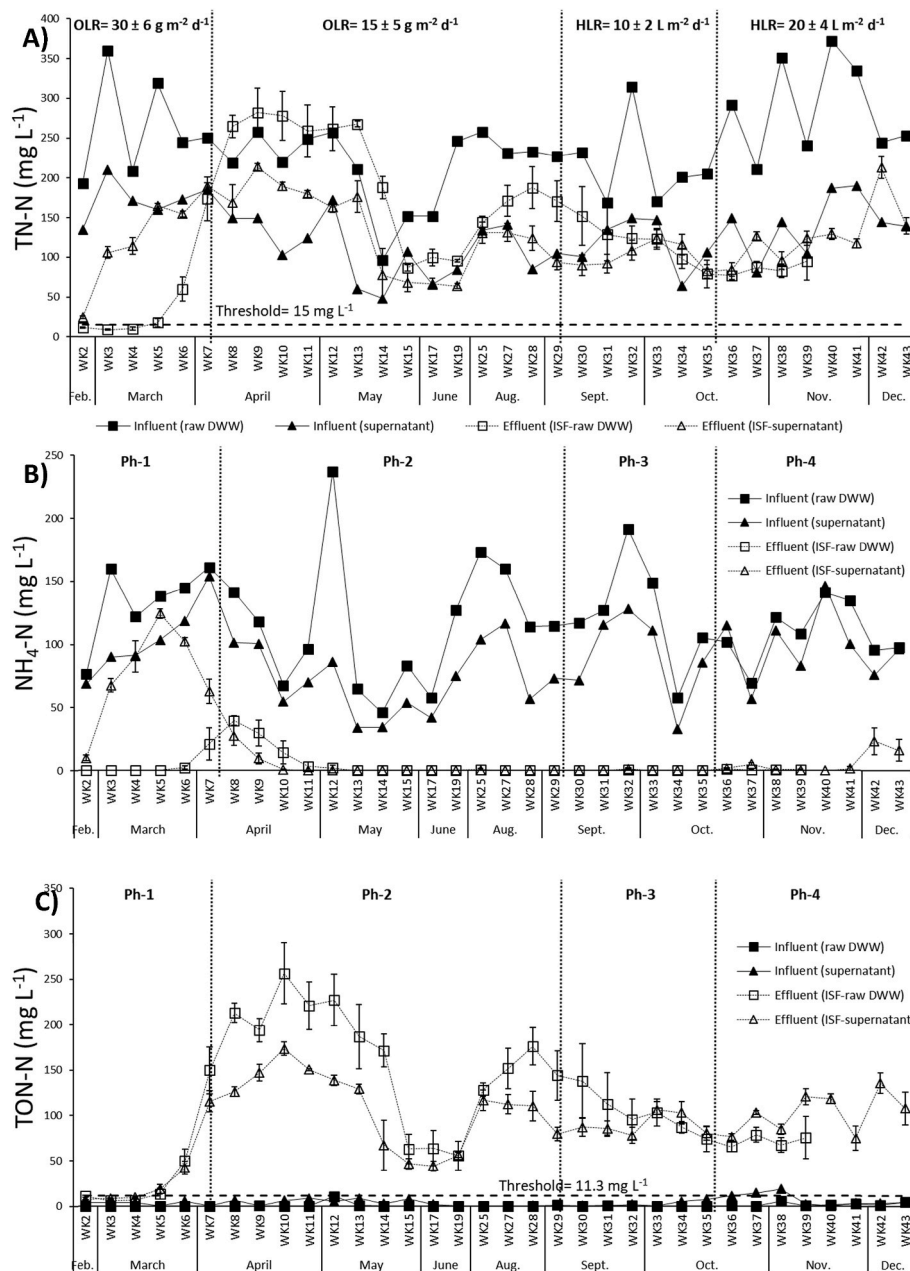


Fig. 6. Influent and effluent concentrations of raw DWW and supernatant filters for a study period of 43 weeks: A) total nitrogen (TN); B) ammonium-N (NH₄-N); and C) total oxidised nitrogen (TON-N).

was higher than supernatant ISFs (Fig. 6-B). In Phase 3, there was no statistical difference in the mean effluent TON-N between raw DWW and supernatant ISFs ($P > 0.05$; Fig. 6-C). During this phase, the effluent TON-N from the raw DWW ISFs started to be lower than those from supernatant ISFs for the first time (Wk 33; Fig. 6-C), although the influent NH₄-N was higher for the raw DWW ISFs than supernatant ISFs, and both sets of ISFs achieved full nitrification. In Phase 4, the effluent TON-N from raw DWW ISFs was lower than supernatant ISFs ($P < 0.05$; Fig. 6-C). This indicated that raw DWW ISFs were partially capable of denitrification in this phase. This coincided with the period when the raw DWW ISFs started to be saturated and clogged, which are major factors in the development of anoxic zones. Chen et al. (2021, b) observed simultaneous nitrification and denitrification in the clogging zones in the top layers of an ISF, and also found that increased moisture in the media may increase denitrification. In addition to the development of anoxic zones in raw DWW ISFs due to clogging, the higher COD

to NH₄-N ratio (46) in the influent raw DWW than influent supernatant (ratio of 15), provided the raw DWW ISFs with the advantages to perform denitrification better than supernatant ISFs. The partial occurrence of denitrification in Phase 4 led possibly to higher TN removal efficiency in the raw DWW ISFs (64%) than the combined coagulation-ISF (53%), as opposed to Phase 2 when the combined system achieved better TN removal efficiency than raw DWW ISFs (52% versus 27%). In general, denitrification was limited for both sets of ISFs in all phases (Fig. 6-C), and the effluent TN from both filters comprised mainly TON-N (Fig. 7-B). Optimal conditions for maximum denitrification occur at a COD to TN-N ratio > 10 (Henze et al., 2008). Both sets of filters achieved this ratio in the current study, thus, it is postulated that using flow recirculation mode to stimulate de-nitrification could bring the TN-N level in the effluent to the standard limit set by the EU directive for urban wastewater discharge (15 mg L⁻¹; 91/271/EEC; EEC, 1991). However, on a few occasions the TN concentration exceeded this

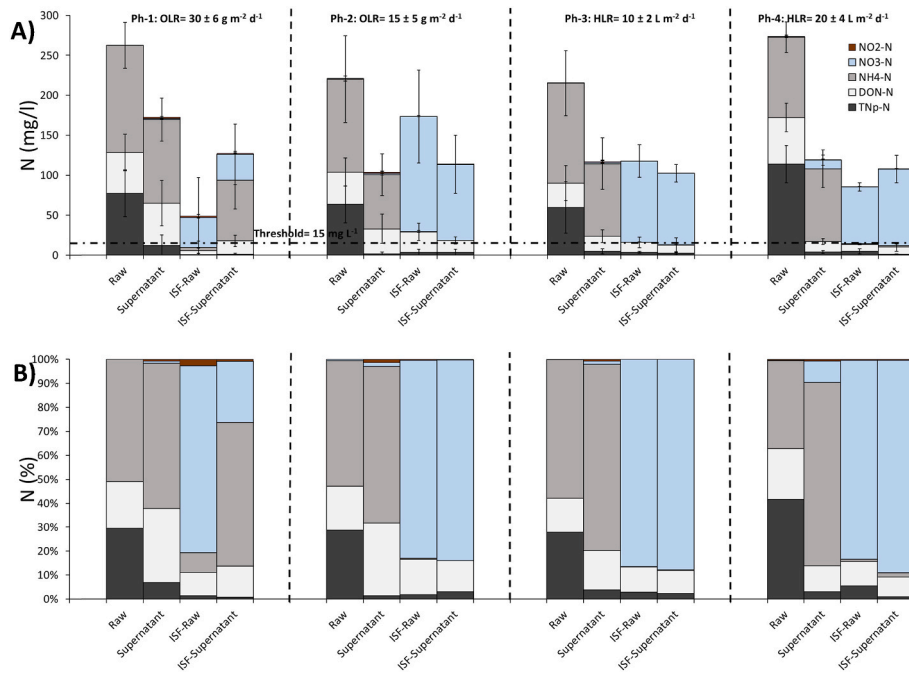


Fig. 7. Mean influent and effluent total nitrogen concentrations of raw DWW and supernatant filters for Phase 1 (n = 6), Phase 2 (n = 11), Phase 3 (n = 6) and Phase 4 (n = 4): A) Total nitrogen fractions in mg L⁻¹ (particulate N (TNp-N), dissolved organic nitrogen (DON-N), ammonium-N (NH₄-N), nitrate-N (NO₃-N) and nitrite (NO₂-N)); B) % of relative total nitrogen fractions.

threshold value by the only combined effluent of DON-N and TNp-N and without TON (Fig. 7-A; Fig. S2-A).

3.5. Volumetric moisture content and clogging status

During Phase 2, the θ_v in the uppermost layer (0–5 cm) for raw DWW filters was similar to that in the supernatant filters (Fig. 8; Fig. S4-A), with no statistically significant differences ($P = 0.62 > 0.05$). In the rest of the layers, the supernatant filter recorded corresponding moisture contents significantly higher ($P < 0.001$) than those recorded in the raw DWW filters. The higher HLR in the supernatant filters (10 L m⁻² d⁻¹) than raw DWW filters (3 L m⁻² d⁻¹) was the main reason of these higher values of θ_v recorded for supernatant filters.

In Phase 3, the θ_v for the uppermost sand layer (0–5 cm) of raw DWW filters increased significantly ($P < 0.001$) to $47.2 \pm 5.7\%$, and was higher than that recorded for the supernatant filters ($31.5 \pm 1.16\%$; $P < 0.001$; Fig. 8; Fig. S4-B). The high OLR and SSLR in raw DWW filters led to this elevated level of moisture content. The differences in θ_v between raw DWW and supernatant filters diminished with depth below the filter surface.

In Phase 4, the θ_v for the uppermost sand layer (0–5 cm) of the raw

DWW filters was significantly ($P < 0.001$) higher than that recorded for the supernatant filters (Fig. 8; Fig. S4-C). Similar trends were observed in the 5–10 cm, 10–15 and 15–20 layers, where in all cases there was a significant difference between the θ_v in the raw DWW and supernatant filters. There was only a small, but significant, difference in θ_v between raw DWW and supernatant filters for the 20–25 cm depth increment, indicating that biofilm build-up due to the increased OLR in the raw DWW filters had abated by that depth. Clogging occurred in the raw DWW filters in Wk 40, but the supernatant filters continued on until the end of the study.

The TOC content in the uppermost layer (0–0.05 m) for the raw DWW ISFs was significantly ($p < 0.001$) higher than the supernatant ISFs, indicating that biomass build-up in the raw DWW ISFs was twice the biomass accumulated in pre-treated ISFs (Fig. 9). The differences in TOC between raw and supernatant ISFs reduced with the depth below the surface (Fig. 9). The high OLR (110 g COD m⁻² d⁻¹) and SSLR (46 g SS m⁻² d⁻¹) in Phase 4 were the main factors that resulted in the raw DWW filters clogging. The high OLR resulted in the accumulation of heterotrophic biomass/microorganisms and secretions of extracellular polymers on surfaces as biofilms, which caused surface sealing (Rodgers et al., 2004). Previously, Leverenz et al. (2009) presented a correlation

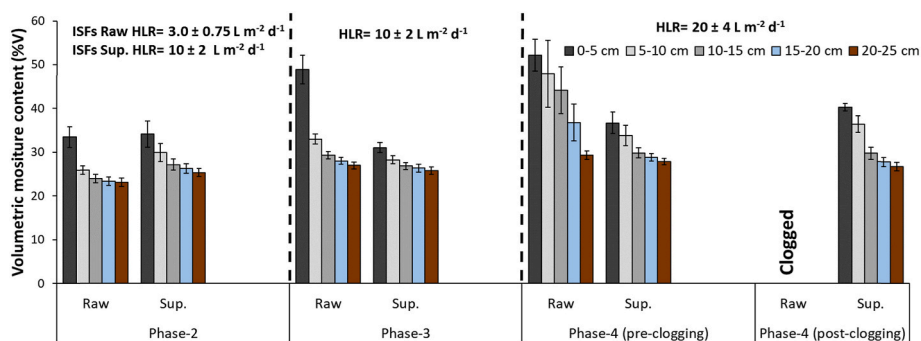


Fig. 8. Volumetric moisture content measured at depths 0–5, 5–10, 10–15, 15–20 and 20–25 cm in: Phase 2 (n = 9), Phase 3 (n = 6), and Phase 4 (pre-clogging n = 4, post clogging n = 4).

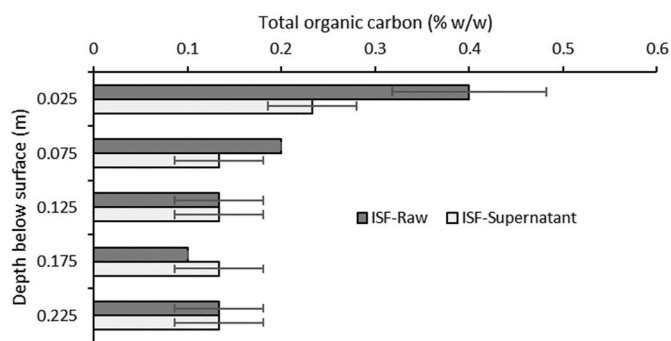


Fig. 9. Total organic carbon (TOC) measured at the end of experiment, at depths 0–0.05, 0.05–0.1, 0.1–0.15, 0.15–0.2 and 0.2–0.25 m.

between SSLR and filter clogging, and estimated $26 \text{ g SS m}^{-2} \text{ d}^{-1}$ to be the upper limit of particulate loading onto ISFs.

Increasing the dosing frequency from 4 to 8 times per day, and increasing the HLR by double in Phase 4, may have accelerated the clogging of the raw DWW filters. Monte Carlo analysis performed by Chen et al. (2021b) indicated that the lifetime of an ISF is negatively related to influent strength and HLR, but is more sensitive to influent strength than HLR. Their study suggested that an increase in HLR can send more biological removal burdens to layers below the clogging development zone, and therefore negatively impact effluent quality. Leverenz et al. (2009) found that ISFs operated at high dosing frequencies, for a given influent COD concentration, have continuous heterotrophic bacterial growth at the surface, a factor in premature failure, whereas loading regimes with low dosing frequencies and/or influent COD concentrations, were found to result in steady-state growth conditions, and therefore long-term, stable operation. The low dosing frequency increases the resting period which allows for endogenous decay and desiccation to partially recover the filter porosity.

3.6. Perspective and implications of the study

3.6.1. Comparison between conventional and hybrid ISF systems

When both sets of ISFs were operated under the same OLR in Phase 2, there was no significant differences in the performance between the two systems, with minor advantages in favour of the hybrid coagulation-ISF system (Table S2). As soon as the conventional ISF system was operated under the same HLR conditions as the hybrid system in Phase 3 and 4, the effluent quality from the raw DWW ISFs declined for most of the measured parameters, and clogging occurred. Overall, the hybrid coagulation-ISFs achieved superior effluent quality with high removal efficiencies ($\% R \geq 99\%$) for all parameters, except for total nitrogen (Table S2). It is likely that TN could be minimized to below the standard limit of 15 mg L^{-1} (91/271/EEC; EEC, 1991) if denitrification was stimulated by effluent recirculation. The performance accomplished by the hybrid ISF system in the present study was better than those attained by conventional ISFs reported in the literature (Rodgers et al., 2005; Torrens et al., 2009; Ruane et al., 2014; Murnane et al., 2016; Table S2).

The surface area of ISFs are usually designed based on OLR. Coagulation pre-treatment reduced the organic matter (COD) by 76%, thereby facilitating the use of a system with a 75% reduction in size. The reduction in size makes the hybrid system an attractive option for farmers as they would prefer to allocate a small proportion of their farmland for waste management. Considering a farm with 100 dairy cows, with each cow producing approximately 33 L d^{-1} (Minogue et al., 2015), and an OLR of $30 \text{ g m}^{-2} \text{ d}^{-1}$ for the ISFs, this would mean that an area of 145 m^2 ($12 \text{ m} \times 12 \text{ m}$) would be required for a hybrid system versus 600 m^2 ($30 \text{ m} \times 20 \text{ m}$) for a conventional ISF system. This means that land requirement, construction and maintenance costs for the hybrid system will be 75% less than conventional ISF system.

Phosphorous breakthrough commonly occurs in conventional ISFs

due to the limited capacity of sand to adsorb P (Table S2). Eliminating P in the upstream treatment by chemical coagulation will significantly reduce the P load on the ISFs, and therefore sustain them for long period without breakthrough as indicated in the current study (prediction >15 years based on the maximum adsorption capacity of P).

Filter clogging is also a limitation of conventional ISFs (Table S2). The pre-treatment step overcame this defect by substantially reducing the OLR and SSLR, which are the main factors responsible for filter clogging. In the current study, the ISFs of the hybrid system were operated for a complete milking season without showing any ponding or evidence of clogging, while the conventional ISFs clogged on Wk 40. Analysis of the filter media in both ISFs (Mohamed et al. unpublished data) showed that the supernatant filters lost only 40% of their initial hydraulic conductivity in the uppermost layer due to biomass build-up, while the raw DWW filters lost approximately 85% of their initial hydraulic conductivity. This indicated that the supernatant filter could be operated for another milking season without clogging.

The decision to select the coagulation method as a pre-treatment step depends on the wastewater properties. For instance, a hybrid ISF system may only be viable if the wastewater mainly contains COD, TSS or TP, because higher reduction of these parameters can be achieved by FeCl_3 (Table 1), and therefore smaller surface areas are required to treat these parameters. In contrast, no saving in treatment area can be expected if the wastewater is only enriched with ammonium (Table 1). While there are many available options of coagulants that can be used for DWW treatment, the use of FeCl_3 in the current study was based on Mohamed et al. (2020), who found that FeCl_3 was the best performing coagulant for DWW treatment among other different coagulants, namely poly-aluminium chloride and aluminium sulphate. The study of Mohamed et al. (2020) took into consideration many aspects to evaluate and rank these chemical coagulants such as treatment efficiency, treatment cost and the volume of generated sludge. In addition to selection of a suitable type of chemical coagulant pH, dose and mixing time are also important design parameters (Mahmoud and Mahmoud, 2021).

3.6.2. Effluent management options for the hybrid system

Considering the large quantity of clean water used daily on farms to wash down holding yards and milking parlours, the effluent from the combined system could be recycled to clean down the collecting yard. Such an option is likely to be more attractive for farmers than a licenced effluent disposal option, not only due to savings incurred due to water usage, but also those costs associated with licensing and regular effluent testing. For urban reuse activities (e.g. landscape irrigation, yard washing, fire protection, cooling systems, etc.), the concentration of the microbial indicators would have to be considered, and final effluent should meet local/national guidelines for water reuse (e.g. U.S. EPA guidelines $\text{pH} = 6\text{--}9$, $\text{BOD} \leq 10 \text{ mg L}^{-1}$, turbidity $\leq 2 \text{ NTU}$, no detectable faecal coli/100 mL; US Environmental Protection Agency, 2004). The faecal indicators were not monitored for this study, but previous studies showed both coagulation treatment and sand filters had good capability to reduce *E. coli*. For example, Mohamed et al. (2020) achieved a high reduction of *E. coli* to below detection limits (about 7.5 log removal) using FeCl_3 to treat DWW. Sand filters can achieve greater than 99.9% (3 log) removal of faecal coliforms (Healy et al., 2007). A simple tertiary treatment method with minimal maintenance such as an activated carbon filter may be used to polish the effluent to comply with US Environmental Protection Agency (2004).

In parallel, the generated sludge, which is enriched with nutrients and P-sorbing coagulants, may have agronomic value as an organic fertilizer, and therefore could be spread on farmland. The formation of ferric-phosphate chemical bonds in the amended sludge will reduce the solubility and mobility of P, thereby minimizing the risk of P losses to water via runoff or drainage (Che et al., 2022).

The cost of coagulation treatment by FeCl_3 was calculated to be $\text{€ } 1.17 \text{ m}^{-3}$ DWW (estimated based on FeCl_3 cost of $\text{€ } 520 \text{ m}^{-3}$). However, 75% of the treated DWW from the hybrid system can be recycled saving

75% of water usage, and hence recover € 1.4 m⁻³ (estimated based on water cost of € 1.87 m⁻³; Irish Water, 2022). The sludge portion (25% of DWW) is also expected to recover some money through a reduction in the chemical fertilizer input. Therefore, coagulation-ISF systems are likely more cost-effective than conventional ISFs, and also have other savings in land, construction and maintenance costs.

4. Conclusion

The hybrid coagulation-ISF system combines the advantages of both technologies, and performs better than a conventional ISF system. On the basis of this study, hybrid systems may be operated at higher HLRs than a conventional ISF systems, and therefore require a smaller sized footprint to treat the same volume of DWW. Unlike conventional ISFs, the hybrid system did not show any signs of clogging or phosphorus breakthrough. The pilot-scale hybrid coagulation-ISF in this study was able to produce high effluent quality (COD: 31 ± 13 mg L⁻¹, TSS: 1.5 ± 2.4 mg L⁻¹, turbidity: 0.39 ± 0.16 NTU, TP: 0.02 ± 0.01 mg L⁻¹, NH₄-N: 0.1 ± 0.09 mg L⁻¹) and achieved removal efficiencies ≥99% for all parameters, except for TN. Future studies should focus on operating the ISFs with effluent recirculation in order to promote denitrification. Future research should also focus on the possibility of reusing the effluent and sludge from the hybrid system for different purposes on the farm.

CRedit authorship contribution statement

A.Y.A. Mohamed: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Validation, Visualization, Writing – original draft, Writing – review & editing. **A. Siggins:** Conceptualization, Funding acquisition, Investigation, Methodology, Supervision, Validation, Visualization, Writing – review & editing. **M.G. Healy:** Conceptualization, Funding acquisition, Investigation, Methodology, Supervision, Validation, Visualization, Writing – review & editing. **O. Fenton:** Conceptualization, Funding acquisition, Investigation, Methodology, Software, Supervision, Validation, Visualization, Writing – review & editing. **D. Ó hUallacháin:** Conceptualization, Funding acquisition, Investigation, Methodology, Supervision, Validation, Visualization, Writing – review & editing. **P. Tuohy:** Conceptualization, Funding acquisition, Investigation, Methodology, Project administration, Resources, Supervision, Validation, Visualization, Writing – review & editing.

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.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jclepro.2022.133234>.

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