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Effect of floating treatment wetland coverage ratio and operating parameters on nitrogen removal: toward design optimization

R. Abi Hanna [a,*], K. E. Borne [b], Y. Andrès [a] and C. Gerente [a]

[a] IMT Atlantique, GEPEA UMR CNRS 6144, F-44307 Nantes, France
[b] National Institute of Water and Atmospheric Research, Private Bag 99940, Viaduct Harbour, Auckland 1010, New Zealand
*Corresponding author. E-mail: r.abihanna@crea-step.fr

RA, 0000-0001-5247-2518

ABSTRACT

Floating treatment wetlands (FTWs) have the potential to improve the quality of wastewater discharges, yet design basics are unavailable to size these systems. This study investigates the effect of FTWs' coverage ratio and hydraulic retention time on agri-food wastewater treatment. This was studied in a pilot-scale experiment comprising four lagoons (6.5 m^3 each) fed with real effluent from an existing tertiary treatment lagoon. An evaluation of FTW of different sizes (L24, L48, and L72 representing 24, 48, and 72% of pilot lagoons surface areas) and a control, L0 (without FTW), was performed over 16 months. Overall, L72 and L48 moderately improved total nitrogen (TN) mass removal compared to L0 ($p < 0.05$), while L24 exhibited similar TN mass removal ($p = 0.196$). The highest improvement was observed for L72, exhibiting up to 55% (mean of 13%) greater N mass removal than the control. The net increase in TN removal by FTWs was mainly related to denitrification, promoted by decreasing dissolved oxygen for increasing FTW coverage ratio. Residence time, temperature, and dissolved oxygen were the main parameters driving TN removal by FTWs. Retrofitting existing lagoons with FTW can facilitate N retrieval through plant harvesting, thereby reducing N remobilization from sediment (common in conventional lagoons).

Key words: design optimization, FTW coverage ratio, plant assimilation, wastewater treatment

HIGHLIGHTS

- Nitrogen removal of floating treatment wetland (FTW) of different sizes was investigated under 16 months at the pilot scale.
- Increasing the FTW coverage ratio to 72% improved total nitrogen (TN) removal by a mean of 12% compared to a conventional lagoon.
- FTW promotes TN removal through denitrification and plant accumulation.
- Plants could accumulate up to 36% of TN removed by FTW.
- Residence time, temperature, and dissolved oxygen were the main parameters driving TN removal by FTW.

1. INTRODUCTION

During the last two decades, floating treatment wetlands (FTWs) have emerged as a promising eco-friendly technology for enhancing wastewater treatment efficiency, particularly for nitrogen and phosphorus removal (Shen *et al.* 2021). FTWs are intermediate derivatives of constructed wetlands (CWs) and ponds. They consist of emergent plants integrated into a floating structure, where roots expand freely into the water body and act as a biofilter for pollutants. FTWs offer a sustainable approach for mitigating nitrogen pollution from various sources through the synergistic interactions between aquatic plants and microorganisms. FTWs are believed to offer potentially more economically attractive alternatives than CWs for retrofitting and enhancing the treatment performance of ponds or lagoons (Wang & Sample 2013). FTWs exhibit reduced footprints (land use) and greater design flexibility than CWs, which require land acquisition for their implementation and heavy civil earthwork. Moreover, FTWs do not present any clogging risk, given that no substrates/filter layers are present as in conventional CWs. Nonetheless, FTW may still show some technical limitations. For instance, they may present variable treatment performances over seasons, as their effectiveness depends greatly on biological processes. They also require periodic maintenance (e.g., plant pruning and removal of accumulated debris) to guarantee good functioning. Moreover, given the limited number of long-term studies and the multitude of floating materials, it is challenging to conclude their aging process and replacement frequencies.

While considerable research has been conducted on FTWs and, more specifically, nitrogen removal, there are notable gaps in the literature that merit further investigation. The absence of design guidelines limits the scalability of FTWs (Bauer *et al.* 2021). Most studies reported in the literature regarding FTW applications for wastewater treatment are conducted in mesocosm with synthetic wastewater and show considerable performance variations. A limited number of pilot and real-scale experiments targeting N removal from wastewater (WW) are found in the literature. Despite encouraging outcomes, comparing results from different studies appears problematic due to the different experimental designs (climatic conditions, plant species and age, inflow concentrations, operating conditions, and FTW coverage ratio) (Lucke *et al.* 2019). Moreover, the existing body of research primarily explores the overall efficiency of FTWs for nitrogen removal, with limited emphasis on the critical role of coverage ratio. For instance, FTWs have been investigated under N concentrations ranging between 0.5 and 59 mg N/L, and they achieved variable total nitrogen (TN) removal ranging from 34 to 85% (Colares *et al.* 2020).

Despite the potential significance of coverage ratio in influencing nitrogen removal, little research has been dedicated to identifying the optimal coverage ratio that maximizes treatment efficiency. Main research reported in the literature explored the effect of plant species, climatic conditions, and water physico-chemical properties on FTW performances through mesocosm and statistical models, providing little representativeness of FTW in real conditions.

This work aims to investigate the nitrogen treatment performance of different-sized FTWs for tertiary agri-food effluents. The present study evaluates, through a pilot-scale experiment receiving real effluent, the effect of FTW coverage ratio and residence time on TN removal. It also explores the relationship between FTW TN mass removal and design and operating parameters. A side-by-side evaluation of FTWs of different sizes compared to a control pilot lagoon (without any FTW) was conducted over 16 months under two residence times. The current study also provides a protocol for screening potential valorization scenarios for the FTW's harvested vegetation to promote a circular economy. Investigated valorization routes include pyrolysis, composting, and anaerobic digestion.

2. MATERIALS AND METHODS

2.1. Experimental design

The present experiment is part of a larger project whose focus also includes customizing the FTWs with a sorbing material to improve P removal. It was extensively described by Abi Hanna *et al.* (2023). The customized FTW modules were each equipped with 5 kg cellular concrete (CC), a phosphorus-reactive material. The present work provides a complementary analysis of the effect of design and operating parameters on N removal under the various design and operating conditions investigated by Abi Hanna *et al.* (2023) and Boonbangkeng *et al.* (under review).

The experimental design consisted of four parallel pilot lagoons installed downstream of an agri-food tertiary treatment lagoon (second lagoon of a series of six tertiary lagoons) to operate with real effluent. The pilot lagoons were designed for 6.5 m^3 capacity (length × width × height: 5 m × 1.3 m × 1.3 m) and were semi-continuously fed maintaining a water depth of 1 m. Effluents exited the pilot lagoons gravitationally through outlet tanks that facilitated water sampling.

The customized FTW module consisted of a molded recycled polyethylene frame (1.3 m × 1.2 m) on which a honeycomb structure was installed to hold the plants. All FTWs were planted with *Carex riparia*, native to northern Europe, perennial, and emergent macrophytes (Ladislas *et al.* 2013) at 10 plants/m^2. The planted FTWs were installed on the real-scale tertiary lagoon for acclimation and establishment in September 2019 and transferred to the pilot lagoons in January 2021.

The effect of 3 FTW coverage ratio (24, 48, and 72%) on TN removal was investigated under 16-day residence time and 400 L/day hydraulic loading rate for 10 months (March–December 2021) (Figure 1). Lagoons equipped with FTW are designated with respect to the applied coverage ratio: L24, L48, and L72 for 24, 48, and 72% coverage ratio, respectively.

The effects of hydraulic residence time (HRT) and hydraulic loading rate on TN removal were also assessed by monitoring the performance of the largest FTW coverage ratio (i.e., 72%) under 8-day residence time and 800 L/day hydraulic loading rate for 6 months (March–September 2022). One pilot lagoon without any FTW served as a control during both experimental periods (L0 and L0′) (Figure 1). During 2022, one pilot lagoon (L72 conv.) was equipped with a 72% FTW coverage ratio without CC to determine whether CC influences TN removal. In this article, data from 2022 were used only to assess the effect of HRT on TN removal; the specific impact of CC is discussed in depth in another paper (Boonbangkeng *et al.* under review).

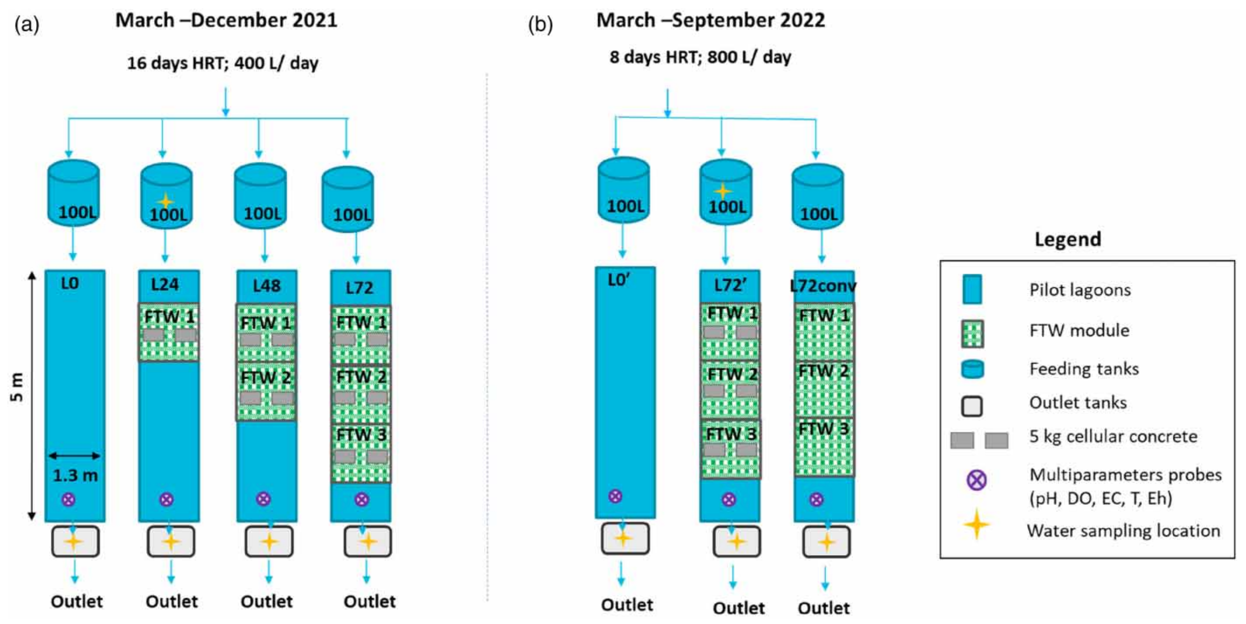

Figure 1 | Pilot experiment setup during (a) 2021 and (b) 2022 monitoring. DO, dissolved oxygen; T, temperature; EC, electrical conductivity; Eh, oxido-reduction potential.

Outflow water pH, dissolved oxygen (DO), electrical conductivity (EC), oxido-reduction potential (Eh), and water temperature were continuously logged during the experiment using *in situ* multiparameter probes (Aqua TROLL® 600 Multiparameter Sonde) installed 45 cm upstream of each pilot lagoon outlet.

Air temperature, solar radiation, and rainfall were also recorded at 15-min intervals using a mini weather station (Watchdog Micro Stations) installed at the experimental site. Meteorological conditions are believed to strongly influence plant growth, biomass density, and microorganism establishment (McAndrew & Ahn 2017) and hence the FTW treatment efficiency.

2.2. Experiment monitoring

2.2.1. Water sampling and analysis

The treatment performance of the pilot lagoons was assessed monthly with flow-weighted composite samples collected over a 24-h period. Samples were collected and preserved according to the Standard Methods For the Examination of Water and Wastewater (2012). All samples were analyzed for total suspended solids, dissolved chemical oxygen demand (COD), biological oxygen demand (BOD_5), ammonium (NH_4^+), nitrate (NO_3^-), total Kjeldahl nitrogen (TKN) by an external certified laboratory (INOVALYS, Nantes). The total-N (TN) concentration was calculated as TKN + NO_3-N, and organic N (Org-N) as TKN – NH_4-N.

Pollutants' mass removal efficiencies (MRE_{24h}) were calculated based on 24-h water sampling as per Equation (1) (Kadlec & Wallace 2008):

$$\text{Pollutant MRE}_{24h}(\%) = 100 \times \left(\frac{V_{in}C_{in} - V_{out}C_{out}}{V_{in}C_{in}}\right) \tag{1}$$

where V_{in} and V_{out} are inflow and outflow volume during the 24-h sampling (L), respectively, and C_{in} and C_{out} are inflow and outflow pollutant concentration of the 24-h flow-weighted composite sample (mg/L), respectively.

Mass removal efficiencies will be presented together with effluent concentrations to gain a comprehensive understanding of the pilot lagoons' efficiency.

Denitrification efficiency was also assessed by a net NO_3-N mass removal efficiency obtained as per Equation (2). This approach excludes N removal/ conversion processes other than nitrification–denitrification and assumes that denitrification is the dominant process associated with N removal (Von Sperling *et al.* 2020). The net NO_3-N mass removed is added to the difference between inlet mass and outlet mass to account for the internal production of NO_3-N (due to nitrification).

$$\text{Net } NO_3 - \text{N MRE}_{24h}(\%) = \frac{m_{\text{TKN,24h removed}} + m_{NO_3-\text{N,24h in}} - m_{NO_3-\text{N,24h out}}}{m_{\text{TKN, 24h removed}} + m_{NO_3-\text{N, 24h in}}} \times 100 \tag{2}$$

where Net $NO_3 - \text{N MRE}_{24h}$ is the pilot lagoon net NO_3-N mass removal efficiency achieved during 24-h monitoring (%); $m_{\text{TKN,24h removed}}$ is the net TKN mass removed from pilot lagoons during 24-h monitoring (g); and $m_{NO_3-\text{N,24h in/out}}$ represent pilot lagoons′ inflow or outflow NO_3-N mass during 24-h monitoring (g).

TKN was used instead of NH_4-N since most organic nitrogen will eventually become NH_4-N (Von Sperling *et al.* 2020).

2.2.2. Plant sampling and analysis

Regular plant assessment was performed to understand N partition and fate within FTW systems and to provide maintenance guidelines for optimal treatment performance.

N uptake by *C. riparia* was determined through analyses of plant tissue sampled quarterly as per Abi Hanna *et al.* (2023). Briefly, N accumulation in plants' roots and shoots was estimated based on plant tissue N concentration (mg/kg) and total biomass (kg) for each FTW module. The total biomass for each FTW module was estimated via biomass measurements and correlation equations developed from shoots' and roots' morphological data (shoot length and width and root network perimeter) and their respective measured dry weight. Coupled with routine water quality analysis, the fraction of N accumulated in plants was estimated as per Equation (3):

$$\%\text{N, plant} = \frac{\Delta m_{\text{N, plants}}}{\Delta m_{\text{N, pilot lagoon}}} \tag{3}$$

where $\Delta m_{\text{N, plants}}$ is the N mass accumulated in plants throughout the experiment monitoring and $\Delta m_{\text{N, pilot lagoon}} = m_{\text{N, in}} - m_{\text{N, out}}$; obtained according to Equation (4):

$$m_{\text{N, in/ out}} = \int_{t0}^{tf} C(t)Q(t)\,dt = \sum_{m=1}^{n} \frac{1}{2}(C_{t_m} + C_{t_{m-1}})(V_{t_m} - V_{t_{m-1}}) \tag{4}$$

where $C_{t_{m-1}}$ and C_{t_m} represent inflow or outflow pollutant concentrations (mg/L) at consecutive sampling times t_{m-1} and t_m, respectively; $V_{t_{m-1}}$ and V_{t_m} represent volume inflow or outflow rate (L/day) at consecutive sampling times t_{m-1} and t_m, respectively; and n is the total number of samples collected during the monitoring period.

In addition, *C. riparia* shoots were assessed for potential valorization scenarios after harvest. Shoots harvested in July 2021 were characterized to determine shoots' dry weight (water content), ash content, elemental composition (CHNS), and calorific/heating value. The shoots' lignin, cellulose, and hemicellulose contents were determined through thermogravimetric analysis. Physico-chemical characterization reference methods and protocols are presented in the Supplementary Material (S0). Moreover, the theoretical biomass methane potential (BMP) of *C. riparia* shoots was estimated using biomass characterization data. While the experimental BMP remains the best information on the behavior of biogas feedstock, theoretical BMP_{max} is a simple and fast feedstock screening method. The maximum theoretical BMP_{max} was calculated as per Ahou *et al.* (2021):

$$\text{BMP}_{\text{max}}(\text{mL } CH_4 \text{ / g volatile solids}) = \frac{1}{100}(A \times C_l + B \times C_P + C \times C_C) \tag{5}$$

where A, B, and C are, respectively, the specific methane yields of lipids, proteins, and carbohydrates expressed in mL CH_4/g volatile solids (VS): ($A = 1.014$, $B = 0.496$, and $C = 0.415$) (Sialve *et al.* 2009); C_l represents substrate lipid concentrations (% D.W basis), and it was assumed to equal 2% in the range of most grass substrates (Mir *et al.* 2006); C_p represents substrate protein concentrations (% D.W basis), calculated by multiplying the TN content (% D.W. basis) of the shoots sample by a factor of 6.25, as described by ISO 16634-1: 2008; and C_c represents substrate carbohydrate concentrations (% D.W basis), equivalent to the sum of cellulose and hemicellulose substrate contents.

2.3. Statistical analyses

Statistical analyses were performed with Minitab 19. All nondetects were replaced by ½ method detection limit as per Von Sperling *et al.* (2020). Data were tested for normality using the Anderson–Darling test. If data were normally distributed, repeated measure analysis of variance (ANOVA) was used to compare the performances of the pilot lagoons and to identify enhancement induced by FTWs and the effect of coverage ratio on TN treatment. Sampling time was set as a random factor, while each treatment was considered a fixed factor. When significant differences between treatments were present (test p-value < 0.05), a Bonferroni pairwise comparison test was performed to identify which pair means differed. When data were not normally distributed, the nonparametric test, Kruskal–Wallis test, was used to compare medians. The effect of residence time on FTW performance was assessed through a one-way ANOVA over L72 and L72′ data.

Moreover, a multilinear regression analysis was developed using experimental data over the 16 months' monitoring period (including 2021 and 2022 monitoring periods) to understand the effect of climate, design, and operating parameters on TN MRE_{24h} achieved by the pilot lagoons equipped with FTW. The main objective was to identify and rank the most influencing parameters to better understand FTWs functioning and inform better design where feasible. The quantitative relationships between the 'dependent variable' TN MRE_{24h} (including data from L24, L48, L72, L72′, and L72 conv.) and 'predictor variables' such as daily mean physico-chemical parameters during the sampling days (pH, DO, EC, Eh, and water temperature), rainfall, mean solar radiation, CC dosage (0, 5, 10, 15 kg), FTW coverage ratio (24, 48, and 72%), and residence time (16 and 8 days) were explored.

Analysis was performed using the stepwise regression model with the backward elimination method with Minitab. The method starts with all the predictor variables in the model and removes the least significant variable for each step ($\alpha > 0.05$). When all the variables in the model had a p-value less than or equal to 0.05, the iteration stopped. Standardized regression coefficients were calculated to compare each predictor variable's relative effect on the dependent variable (i.e., TN MRE_{24h}).

3. RESULTS AND DISCUSSION

3.1. Tertiary inflow composition

TN concentration and the ratio of each form of nitrogen (organic, ammoniacal, and nitrate) at the inlet of the pilot lagoons reflected those of the real tertiary lagoon of the agri-food site from which the effluent was pumped to feed the pilot lagoons. Although TN concentrations were of the same magnitude during the experiment, averaging 11.6 ± 7 and 9.1 ± 3.4 mg/L from March to December 2021 and from March to September 2022, respectively, the ratios of each form of N were different.

As a general trend, N in tertiary effluents was mainly present as NH_4-N, with NH_4-N concentrations increasing in the summer of 2021 (Supplementary Material S1). This might reflect a change in the agri-food effluent composition and/or upstream treatment. Increased ammonification rate and microbial mineralization of organic matter in the upstream real-scale lagoon (first lagoon) due to higher temperatures might be partly responsible for this increase over the summer (Barco & Borin 2017).

The NO_3-N concentrations were relatively low compared to TN concentrations, accounting for $3 \pm 2\%$ and $18 \pm 16\%$ of TN during both monitoring periods (2021 and 2022). While inflow NO_3-N concentrations were 50% of the time lower than the quantification limit (0.2 mg N/L) between March and December 2021, they exceeded this value (78% of the time) between March and September 2022 (ranging from 0.4 to 5.7 mg/L).

Inflow organic N concentrations slightly varied along the experimental monitoring (up to $24 \pm 8\%$ of TN during 2021 and $27 \pm 10\%$ of TN in 2022), with relatively higher concentrations in summer 2021 (up to 5 mg N/L, Supplementary Material S1).

3.2. Effect of FTW coverage ratio on TN removal

During the start of the monitoring in 2021 (first 2 months), TN removal was significantly lower than the rest of the monitoring period. However, it progressively increased following the temperature increase (Figure 2(a)). The first 2 months of operation (May–April 2021) were characterized by cold to moderate mean air temperatures ranging between 2.5 and 27.5 °C (median of daily mean temperatures 13.4 °C) (Figure 2(b)). The water temperatures <15 °C during the startup of the experiment might have slowed the establishment of the system as biological activities are highly influenced by temperature, and their metabolism is known to be inhibited for temperatures below 10 °C (Kadlec & Wallace 2008).

Overall, between March and December 2021, all pilot lagoons ensured TN outflow concentrations lower than the discharge limit (10 mg N/L), as set by French Decree 28 February 2022, except on July 2021 (L72: 11.6 mg/L) (Figure 2(b)).

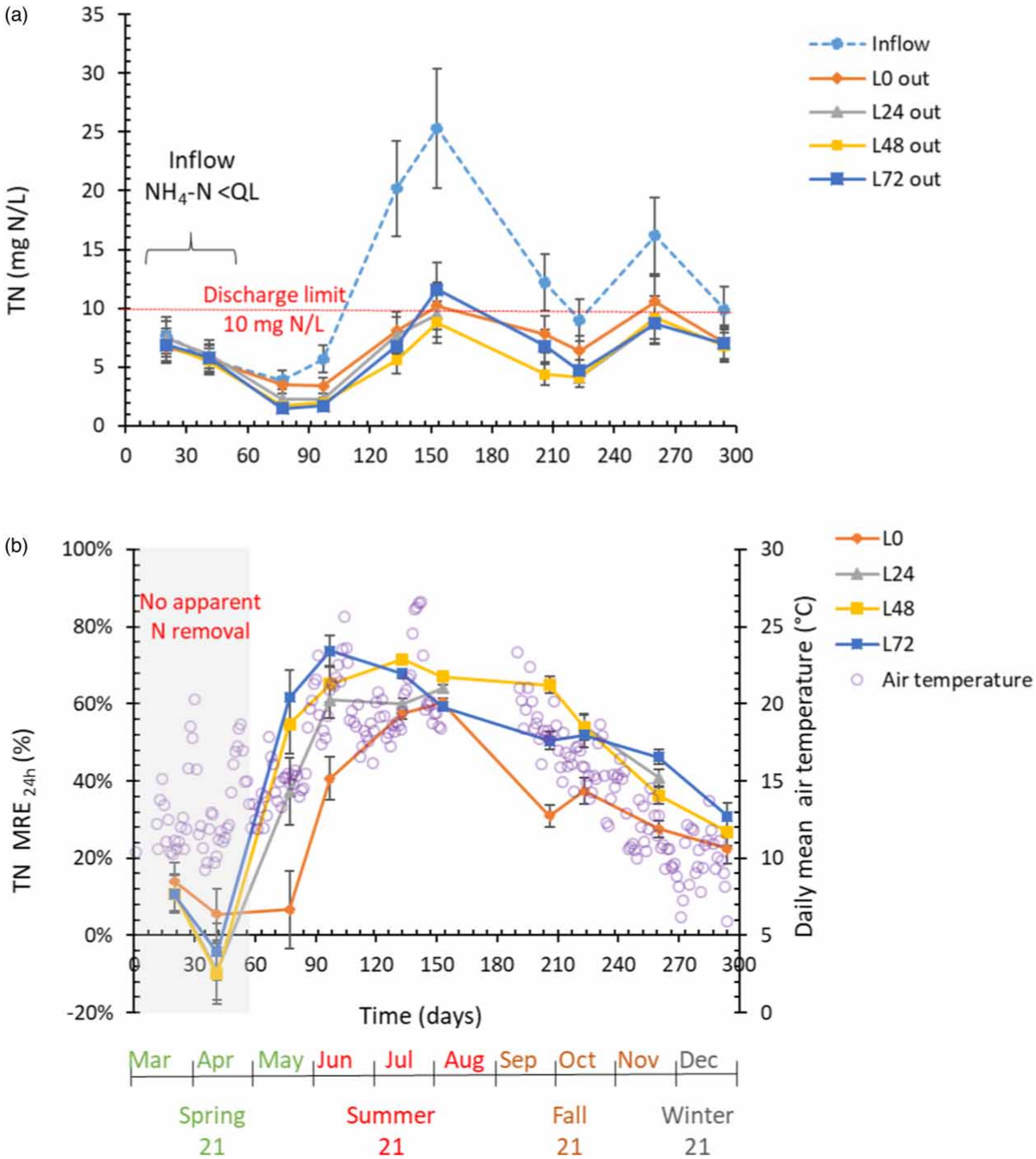

Figure 2 | Pilot lagoons' inlet and outlet TN concentrations (a) and TN mass removal efficiencies (MRE_{24h}). (b) QL, quantification limit.

In terms of mass removal, L24, L48, and L72 achieved 56 ± 9%, 55 ± 16%, and 55 ± 13% mean TN $MRE_{24h,}$ respectively, compared to 39 ± 14% by L0 between May and December 2021 (Figure 2(b)). All FTW coverage ratios appeared to increase TN MRE_{24h} compared to L0: up to 55% TN MRE_{24h} increase was achieved by FTW pilots, which was on average 13% greater than L0 TN MRE_{24h}. However, only L48 and L72 TN MRE_{24h} were significantly higher than that achieved by L0 ($p = 0.024$ and $p = 0.016$, respectively), while L24 and L0 TN MRE_{24h} were not statistically different ($p = 0.393$) (Supplementary Figure S2.1). The limited number of samples collected for L24 (five samples versus eight samples for L0, L48, and L72) would have reduced the statistical test power.

The present study results revealed that increasing the FTW coverage ratio from 24 to 72% improved TN mass removal but to a moderate extent. This is not entirely aligned with the previous findings of Garcia Chance & White (2018) and Samal *et al.* (2021), where the effect of coverage ratio on pollutant removal from runoff and wastewater was investigated with synthetic wastewater and in mesocosms (short-term experiments), respectively. Both studies suggested that increasing the coverage ratio could substantially improve pollutant removal. The present pilot-scale experiment run under semi-continuous flow and real effluents suggests that increasing the FTW coverage ratio would only moderately promote higher N removal.

3.3. Effect of FTW coverage on N speciation

Pilot lagoons' inflow and outflow N forms were investigated in parallel to physico-chemical parameters (DO and pH) near the outflow to better understand the effect of FTW coverage ratio on TN removal processes and pathways.

3.3.1. Influences on NH_4 and NO_3 removal

All pilot lagoons significantly reduced inflow NH_4-N concentrations ($p = 0.035$) and exhibited higher NO_3-N outflow concentrations than their inflow concentrations (Supplementary Figure S2.2). The simultaneous decrease of NH_4-N and increase of NO_3-N concentrations suggest efficient nitrification within all pilot lagoons despite the low DO concentrations recorded in L48 and L72 (Supplementary Figure S2.2). Indeed, roots are believed to provide aerobic sites in the overall low DO environment induced by the FTWs, which can maintain nitrification activity (Pishgar *et al.* 2021). Headley & Tanner (2012) also reported that nitrification was sustained in oxygenated zones and at the interface of oxygen-saturated and unsaturated zones in wetlands.

NH_4-N outlet mass loads appeared to be relatively greater for L72 than the other lagoons, suggesting the lower rate of nitrification for the highest coverage ratio. However, no significant statistical difference was found between the NH_4-N outlet loads of the four pilot lagoons (Figure 3(a)). The limited number of collected data may have prevented detecting the effect of the FTW coverage ratio on nitrification.

FTW pilots generally achieved lower outlet NO_3-N mass load as the FTW coverage ratio increased, showing an evident effect of FTW coverage ratio on N removal pathways, particularly on denitrification activity (conversion of NO_3-N into N_2 gas). Between day 90 and the end of the monitoring (May–December 2021, where TN mass removal was significant), L72 achieved significantly lower mean outlet NO_3-N concentrations resulting in 1/3 lower mean NO_3-N discharge mass loads compared to L0 ($p = 0.028$) (Figure 3(b)). Nonetheless, no statistical difference was detected between L72–L24 ($p = 0.509$) and L72–L48 ($p = 1.00$), L48–L0 ($p = 0.103$), and L24–L0 ($p = 1.000$) mean discharge loads.

The net NO_3-N MRE_{24h} highlights the enhanced denitrification within L48 and L72. Indeed, L72 and L48 exhibited the highest net NO_3-N MRE_{24h} averaging $92 \pm 6\%$ and $86 \pm 7\%$ between May and December, resulting in a 23% median net increase compared to L0 (Supplementary Figure S2.1). When comparing the different lagoons' net NO_3-N MRE_{24h}, L72 was significantly different from L48 ($p = 0.017$), L24 ($p = 0.003$), and L0 ($p = 0.014$), while L48 was significantly different from L0 ($p = 0.043$) (and L72 as stated previously). L24 achieved $79 \pm 7\%$ average net NO_3-N MRE_{24h} and was not significantly different from L0 (net NO_3-N $MRE_{24h} = 54 \pm 23\%$) ($p = 0.075$). These results suggest that the anoxic DO concentrations in L72 and L48 would have promoted advanced NO_3-N removal through denitrification (Supplementary Material S2). Plants would have also contributed to additional NO_3-N removal by assimilation, given that NO_3-N is the preferential N form known to be taken up by plants (Vymazal 2008).

As the increasing coverage ratio is found to ensure improved TN removal, care must be provided to avoid degrading the physico-chemical properties of the water column. The increase in the FTW coverage ratio likely depleted water DO concentrations due to the increased physical barrier limiting oxygen exchanges with the water column. Moreover, an increased FTW coverage ratio is believed to promote shading and limit algal photosynthesis, which is usually responsible for increased DO during daytime and greater organic release through the roots, supporting microbial activity that consumes DO (Borne *et al.* 2014). Hence, if the FTW coverage ratio is to be increased to improve N removal (e.g., up to 72%), it is important to ensure effluent reoxygenation before discharge into the water body, as DO concentrations <4 mg/L would endanger aquatic life.

3.3.2. Influences on organic N removal

The effect of FTW coverage ratios on N pathways was also highlighted through the difference in organic N removal. L72 organic N MRE_{24h} were significantly higher than L0 MRE_{24h} ($p = 0.002$) (Figure 4(a) and 4(c)). However, despite some organic N release from L0 due to algal senescence intrinsic to open water surface lagoons (Abi Hanna *et al.* 2023), no significant difference was found between L24 and L0 ($p = 0.109$) and L48 and L0 ($p = 0.320$).

In general, pilot lagoons' outflow organic N concentrations only slightly varied over the monitoring period (except for L0 in November), even when the organic N concentration in the inflow increased, reaching 5 mg N/L between June and August (Figure 4(a)). L0 high organic N outflow concentration observed in November 2021 most probably reflects algae senescence and decomposition, as indicated previously by Abi Hanna *et al.* (2023). The mean organic N concentrations at the outlet of all

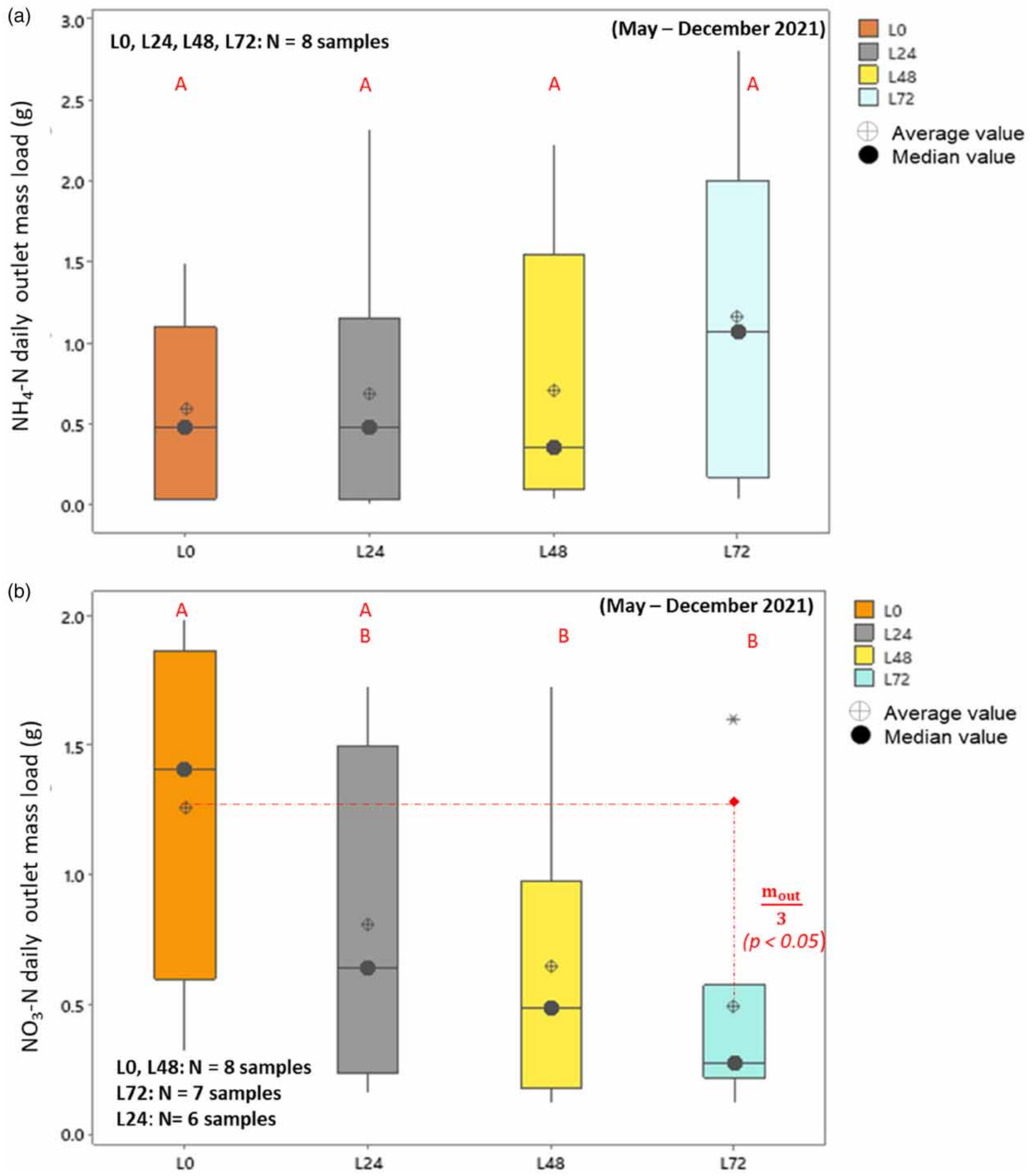

Figure 3 | Box plot of pilot lagoons' (a) NH_4-N and (b) NO_3-N daily outlet mass loads between May and December 2021 (no NO_3-N removal was observed between March and April 2021). The box shows the 25th, 50th (median), and 75th percentiles, and the whiskers represent the maximum–minimum range. Note: * Outlier. Means that do not share a letter are significantly different.

pilot lagoons averaged 2.2 ± 0.5 mg/L and were within the typical organic N background concentration range in free water surface wetlands (1.5–2 mg/L) (Kadlec & Wallace 2008).

3.4. Role of FTW in sustaining denitrification

The higher organic nitrogen MRE_{24h} and COD MRE_{24h} achieved by L72 compared to L0, L24, and L48 may reflect the extent of denitrification observed within this lagoon (Figure 4(b) and 4(d)). In L48 and L72, a larger amount of plants could have provided greater quantities of endogenous carbon sources as they are known to secrete and leach carbonaceous compounds from their roots (Zhai *et al.* 2013), adding to it the additional C from decaying plant biomass. Indeed, the limited inflow COD

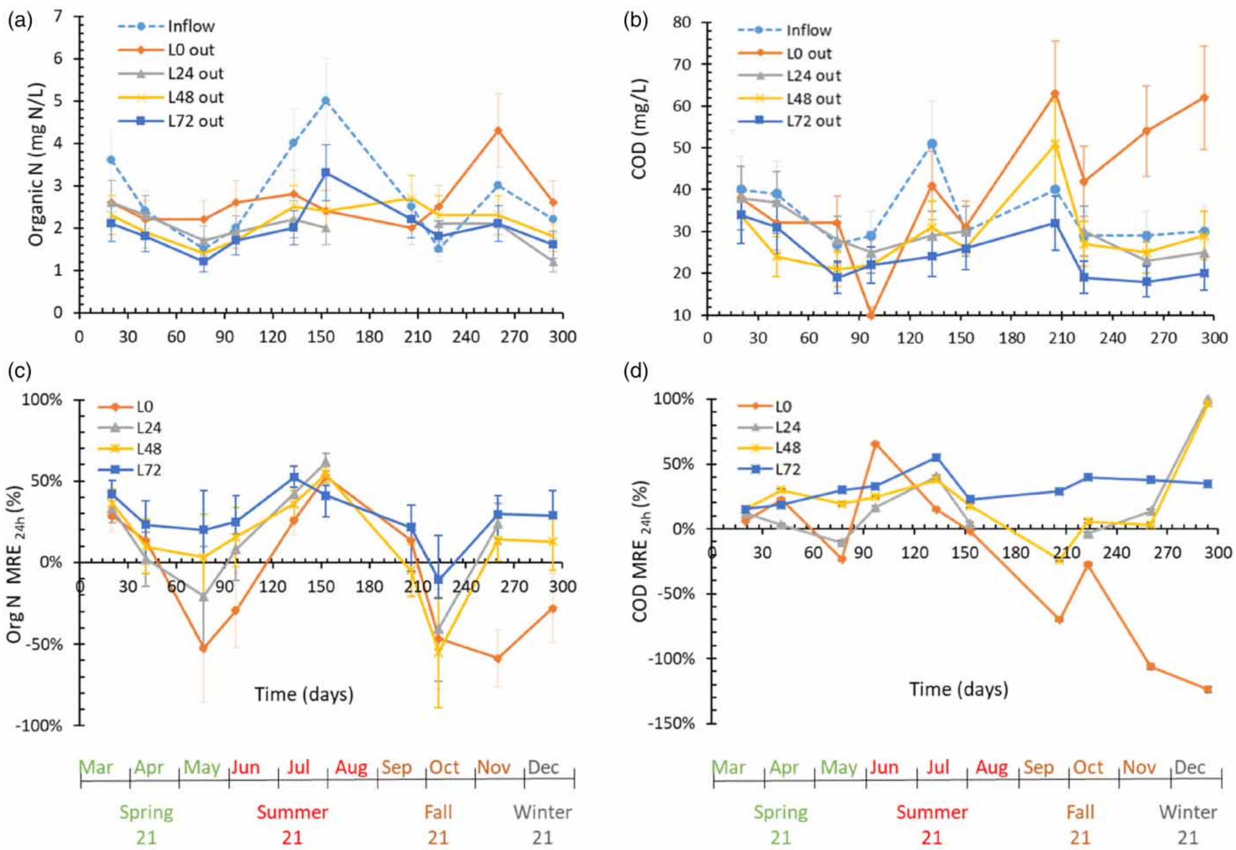

Figure 4 | L0, L24, L48, and L72 inlet and outlet: (a) organic N and (b) COD concentrations and their MRE_{24h} (c and d) during the 2021 monitoring.

concentration (34.4 ± 7.7 mg/L mean inflow concentration), suggesting an even lower source of biodegradable organic carbon in the influent, may have hindered denitrification in L0 and L24 under prevailing aerobic conditions.

While carbon mass balance could not be estimated in the present study, the hypothesis that plant exudation may have sustained denitrification is consistent with the findings of Lynch & Whipps (1990). The authors reported that 30–70% of net carbon fixed by photosynthesis would be translocated to root tissue, of which 40–90% is used by plant roots for respiration or released into the rhizosphere. Similarly, Zhai *et al.* (2013) estimated that root exudation in phytoremediation processes could fuel the denitrification rate by up to 26–73 mg/m^2 wetland/day.

3.5. Role of plants in N removal

The plant's N accumulation within the FTW accounted for only 7 and 11% of TN removal in L24 and L48, suggesting that it was not the main removal pathway (Figure 5). However, in L72, plant accumulation contributed up to 24% of TN removal. Indeed, the plant contribution to overall N removal is influenced by planting density (coverage) and design parameters (e.g., loading rates and plant species, accumulation capacity) (Sharma *et al.* 2021). For instance, in studies addressing FTW performances, substantial variability is found among plants' contributions to overall N removal. Plants' contributions to TN removal by FTW range from insignificant to up to 74% of TN removed by FTW. However, the reported results in the literature are heterogeneous since they are obtained with different plant species, rendering comparisons between the numerous coverage ratios complex (Colares *et al.* 2020). In our study, the plant contribution to TN removal was relatively replicable between pilot lagoons equipped with same-sized FTWs. *C. riparia* could take up to 28.6 ± 5.2% of TN removed by 72% coverage ratio FTW (mean of the plants' contribution to TN removal in L72, L72′, and L72 conv.).

On average, up to 27 g N/m^2 FTW (N removed/ FTW module surface area) was removed from tertiary effluents using *C. riparia* at 10 plants/m^2 (between March and September of 2021 and 2022). The comparable plant uptake rate was

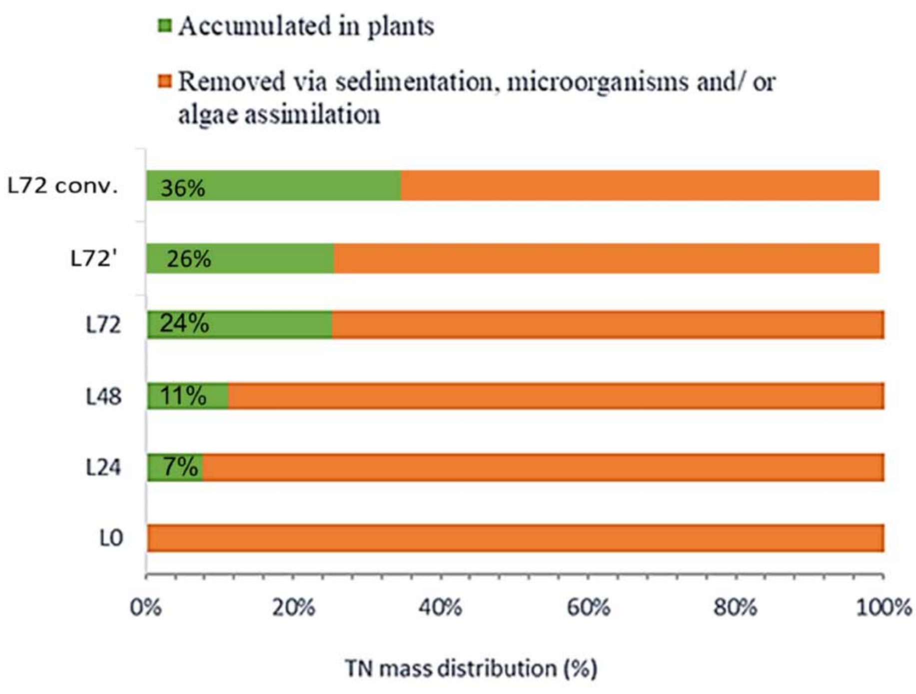

Figure 5 | Plant contribution to overall TN mass removal within pilot lagoons.

found with *Carex apressa* used in a field experiment to remove nutrients from a waste stabilization pond (Huth *et al.* 2021). Under 9 mg/L TN inflow concentration and 3 plants/ m^2 plant density, 22 $\pm$ 6 g/m^2 was accumulated in *C. apressa* shoots during 1 year of operation producing 0.91 $\pm$ 0.07 kg shoots/m^2 (Huth *et al.* 2021). Based on these observations, a strategic plant harvest might be beneficial to prevent N release into the water body during plant senescence. In the present experiment, pruning the *C. riparia* plants in autumn promoted vigorous shoot regrowth, suggesting that it could also promote N sequestration during the next growing season. The harvested biomass could be redirected and used for nutrient recovery or energy production within a circular economy approach. The physico-chemical analysis of harvested biomass provides insight into their potential valorization routes and will be the scope of Section 3.7.

3.6. Most influencing parameters on N removal

The unstandardized multilinear regression equation obtained with FTW data from the two monitoring periods (2022 and 2021) showed that FTW TN MRE_{24h} was positively correlated to the residence time and mean water temperature and negatively correlated to mean DO concentration (Equation (6)):

$$\text{TN MRE}_{24\text{h}}(\%) = -0.496 + 0.032\,\overline{\text{wTemp}} + 0.037\ \text{RT} - 0.05\,\overline{\text{DO}}\,,\quad R^2 = 87\%,\ \ \text{N} = 31 \tag{6}$$

where RT is the retention time in days (16.25 or 8 days); $\overline{\text{DO}}$ is the mean dissolved oxygen concentration during sampling day; and $\overline{\text{wTemp}}$ is the mean water temperature during sampling day.

DO concentration was strongly correlated to FTW coverage ratio as detailed by Abi Hanna *et al.* (2023). Hence, the DO in the regression equation also stands for the effect of the FTW coverage ratio on TN mass removal. Indeed, by increasing the FTW coverage ratio, DO concentrations were reduced, favoring TN removal through denitrification (Boonbangkeng *et al.* under review).

The regression-independent variables explained 87% of the FTW pilots' TN MRE_{24h} variability (Figure 6). Four experimental points (out of the 35 water sampling missions) showed high residuals and were excluded from the regression. These data points corresponded to sampling days where TN MRE_{24h} was low compared to the mean TN MRE_{24h} obtained during the rest of the monitoring period. The reasons behind these performances remained unidentified.

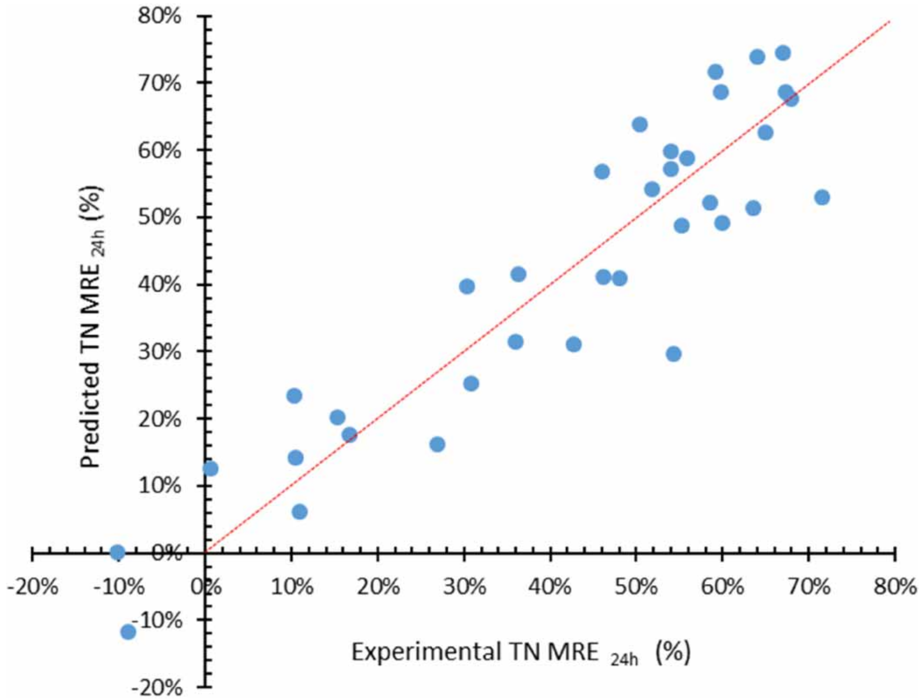

Figure 6 | FTW TN MRE_{24h} predicted by the multilinear regression model versus experimental TN MRE_{24h}.

The regression analysis did not include cellular concrete as one of the most influencing factors on TN MRE_{24h}. This result is in agreement with the findings of Boonbangkeng *et al.* (under review), where cellular concrete provided a limited increase in FTW TN removal compared to a conventional FTW (without cellular concrete). The water column pH was relatively consistent, close to neutrality, and similar in all pilot lagoons equipped with FTW and cellular concrete (L72′) and FTW alone (L72 conv.) (Figure 7, data collected during the second monitoring period in 2022). Meanwhile, pH showed very sporadic behavior in the control lagoon (L0′), most probably impacted by algal photosynthesis responsible for increased DO concentrations. Despite the alkaline nature of CC present in L72′, similar water column pH in L72′ and L72 conv. suggests a buffering effect of the floating wetland keeping pH between 6.8 and 7.2 during the whole monitoring period (Figure 7). This behavior

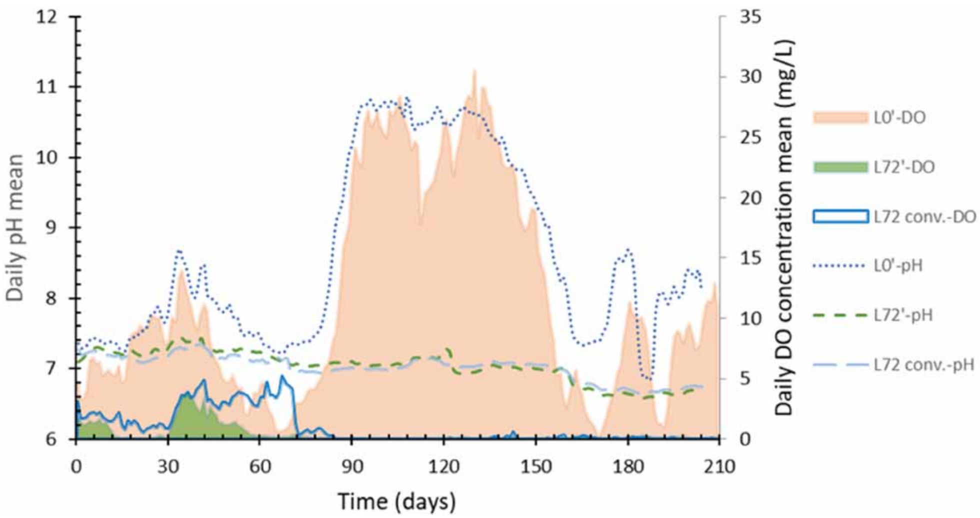

Figure 7 | Pilot lagoons′ daily pH and dissolved oxygen (DO) mean concentrations upstream pilot lagoons′ outlet during 2022 setup.

may be the result of simultaneous consumption of OH^- driving phosphate removal by CC, as described by Abi Hanna *et al.* (2023). Indeed, the CC was added to FTW to promote phosphate removal by sorption and precipitation. The buffered pH could also be the result of interaction of OH^- released by CC with acidic exudates of roots (consuming alkalinity), of greater importance in dense FTW (72% FTW coverage ratio). The stable pH prevailing in pilot lagoons equipped with FTWs is believed to favor biofilm survival and sustain simultaneous nitrification–denitrification, compared to sporadic pH ranges in control lagoon.

While mean water temperature appeared to have the most significant effect on TN MRE_{24h} (standardized coefficient: 0.18), residence time exhibited the second most significant effect on TN MRE_{24h} (standardized coefficient: 0.16), followed by the mean DO concentration (standardized coefficient: −0.13).

However, when comparing L0′ and L72′ TN MRE_{24h} (21 ± 21 and 40 ± 18%, respectively) (Boonbangkeng *et al.* (under review)) to L0 and L72 (39 ± 14 and 56 ± 9%, respectively), no significant difference between the achieved TN MRE_{24h} was detected. It could be hypothesized that the limited number of data collected during the monitoring periods may not have allowed capturing the effect of HRT through comparative statistics (i.e., one-way ANOVA over L0, L0′ and L72, and L72). More extended monitoring periods (more data points) would be needed to confirm the HRT effect on TN removal and investigate its interaction with other operating conditions.

From a design perspective, the results of the present study suggest that while an increased FTW coverage ratio promoted increased TN removal (especially up to 72%, showing statistically significant improvement in the present study), the performance improvement remained relatively moderate (average of 13%) when compared to an uncovered lagoon. This finding may be explained by underlying treatment processes, including increased nitrification, algal development, and N uptake, occurring in uncovered water. However, a significant impact of the coverage ratio lies in the removal and distribution of nitrogen forms. The increased coverage ratio promotes anoxic conditions and, therefore, NO_3 removal. Borne *et al.* (2015) suggested that the FTW size likely impacts the intensity of DO depletion directly below the FTW, while the overall coverage ratio (i.e., reflective of how confined the FTW is within a pond) would impact DO depletion beyond the FTW footprint. If denitrification is the preferred removal mechanism, it is recommended to favor one large FTW rather than several dispersed FTWs of equivalent size to develop anoxic conditions underneath the FTW. Indeed, in the case of a 'dispersed layout of smaller FTWs', a greater proportion of FTW edges would be in contact with open water areas, promoting more exchanges with surrounding DO in open water, therefore limiting denitrification.

TN MRE_{24h} of the FTW pilot lagoons were in the same range as those obtained by Van de Moortel *et al.* (2010) with *Carex* spp to treat wastewater of slightly higher N concentration in a mesocosm experiment (TN: 22 mg N/L, NH_4: 16 mg N/L), where FTW achieved 42% TN removal efficiency. However, ammonium removal efficiency exceeded that reported in the work of Van de Moortel *et al.* (2010), where the 100% coverage ratio of *Carex* spp achieved only 35% NH_4-N removal compared to 62 ± 36% in L24, 66 ± 36% in L48, and 57 ± 31% in L72. Ammonium removal could have been limited by the 100% FTW coverage in the work of Van de Moortel *et al.* (2010), potentially decreasing water DO and, therefore, nitrification.

Compared to the work of Wang *et al.* (2020), where the performance of enhanced FTW through the addition of zeolite and sponge iron was assessed over 2 years of a pilot-scale experiment, the current FTW ensured comparable TN removal. The present study results showed that FTWs maintained outflow concentration <10 mg N/L discharge limit without the need to incorporate a specific ammonium sorbing material. Wang *et al.* (2020) suggested that a 45% coverage ratio could achieve 61.5% TN concentration reduction at 10 plants/m^2 and almost 8 kg/m^3 filter material under 8.2 ± 1.7 mg N/L mean TN inflow concentration. In the present study, FTW with only a 24% coverage ratio and 10 plants/m^2 achieved 55 ± 13% TN removal (under 11.6 ± 7 mg N/L mean inflow concentrations). These results emphasize the great potential of FTW in providing additional N treatment without the need for any N sorbing material.

3.7. Fate of harvested biomass

Under the current operation conditions, harvested shoot biomass reached on average, 2.7 ± 0.4 kg D.W./FTW module in October 2021. It slightly increased in September 2022 to 3 ± 1 kg D.W./FTW (shoot production following the harvest at the end of the first experiment in 2021). Compared to other common plants used in FTW for wastewater application, Barco *et al.* (2021) reported that *C. riparia* seems to produce high mean shoot biomass (averaging 2.4 kg/m^2) compared to 1.5 kg/m^2 by *Typha*, 1.3 kg/m^2 by *Phragmites*, and 1.1 kg/m^2 by *Iris*.

The low heating value (LHV) of *C. riparia* and ash content (8.5 ± 0.1%) may suggest its suitability for thermochemical valorization (feedstock ash content for thermochemical valorization <20%). However, the shoots' high water content (low dry weight, 32%) could impact the energetic yield. Indeed, higher water content would increase the energy consumption to dry the feedstock before thermal treatment (i.e., pyrolysis).

On the other hand, the harvested biomass presented a C/N ratio equal to 18, which is in the range of optimal C/N ratio for composting (10–30) and/or anaerobic digestion (10–90) (Ahou *et al.* 2021). Shoots of *C. riparia* had a similar average composition to that obtained for the wetland plants studied by Marchetti *et al.* (2016), except for remarkably lower dry weight (Table 1). While composting appears to be the most common and easy valorization for the harvested biomass, the low lignin content (13 ± 1%) suggests potential for anaerobic digestion. This is also supported by *C. riparia* shoot theoretical biomass methane potential (BMP_{max}) of 301 mL CH_4/g VS, which is in the range of BMP of 23 wetland plant species investigated by Marchetti *et al.* (2016). The investigated plants comprised *C. riparia, Arundo donax, Cynadon dactylon, Scirpus selavticus*, and *Phragmites australis* and exhibited mean BMP equal to 213 mL CH_4/g VS (Marchetti *et al.* 2016). Although the practical efficiency obtained in a biogas reactor is expected to be lower than the theoretical biomethane potential, *C. riparia* shoots appear to be relatively suitable for methane production as its BMP was higher than that obtained from agro-industrial waste (Dinuccio *et al.* 2010). The *C. riparia* biomass could be used in co-digestion with other waste materials to increase biomethane production. Moreover, if biomass is to be directed for anaerobic digestion, care must be provided to ensure a low lignin content. Pereira *et al.* (2022) found that shoots could present lower lignin content when a single harvest or two harvests are performed in late summer or at early growth stages. Accordingly, it is crucial to determine the harvest period in advance based on the valorization scenario retained for the harvested biomass.

4. CONCLUSION AND PERSPECTIVES

The present study consisted of a FTW pilot-scale experiment representative of a real-scale application for tertiary agri-food wastewater treatment. Three FTW coverage ratios were assessed for their effect on TN removal. While all FTW coverage ratios were found to ensure satisfactory TN mass removal (~50%), a limited N removal increase was found compared to the uncovered lagoon (13% average increase), and it was achieved by the 72% FTW coverage ratio. The TN removal is believed to increase with the FTW coverage ratio. Based on the present study, a minimum of 48% FTW coverage ratio is needed to obtain significant TN removal enhancement compared to conventional lagoons. This is mainly related to the effect of coverage ratio on DO concentration, which impacts TN removal processes. Indeed, one of the most significant

Table 1 | *Carex riparia* shoots physico-chemical characterization

Physico-chemical properties	Present study	Marchetti *et al.* (2016)
Dry weight (%)	32%	94.7 ± 1.0%
Ash content (%)	8.5 ± 0.1	11 ± 4.9
Carbon (%)	42 ± 0.2	42.9 ± 2.4
Hydrogen (%)	5.8 ± 0.2	n.d
Nitrogen (%)	2.3 ± 0.1	2.3 ± 1.2
Sulfur (%)	<0.06	n.d
Protein calculated (%)[a]	14.4	14.4
C/N ratio	18	26 ± 19
Lignin (%)	13 ± 1	13.4 ± 7.2
Cellulose (%)	20 ± 4	23.6 ± 12.7
Hemicellulose (%)	33 ± 0.7	19.2 ± 19
HHV (MJ/kg D.W.)	17.7 ± 0.5	n.d
LHV (MJ/kg D.W.)	14 ± 0.5	n.d

Note: All data are given as a percentage of dry weight (D.W.), the dry weight is expressed as a percentage of fresh weight, C/N is unitless. Biomass average composition of 23 wetland species.
n.d, not determined; HHV, high heating value; LHV, low heating value.
[a]Calculated from N concentration: protein (%) = 6.25 × N (%).

impacts of the FTW coverage ratio in the present experimental setup was the influence on the predominant N removal pathways compared to a conventional lagoon. Increased FTW coverage ratio promoted greater accumulation in plant tissues, making retrieving N from the system easier through shoot harvesting. It also increased denitrification compared to an uncovered lagoon (reducing NO_3 release to the receiving environment). These results are promising as N is mainly removed through sedimentation and microorganisms/algae assimilation in a conventional lagoon, inducing greater accumulation in the sediment and potential subsequent long-term release in the water column. FTW inclusion in existing tertiary treatment lagoon could benefit receiving environments highly sensitive to nitrate input and, from a maintenance perspective, would promote easier N retrieval (compared to sediment/decaying algae dredging), reducing the risk of N release. Moreover, FTW could be a beneficial and feasible retrofit for existing lagoons where land acquisition is difficult and nitrogen discharge limits are stringent. N removal in FTW is mainly driven by biological processes (nitrification and denitrification, plant assimilation), which depend highly on temperature. At temperatures lower than 15 °C, these processes could be significantly hampered. Hence, precaution should be taken when transposing the current study results to a cold climate as MRE are believed to be reduced.

To optimize FTW design and determine the best design combination, a mechanistic and/or compartmental model approach should be considered in the future, which separates the open water body and FTW reactions or treatment behavior. Such a model may allow, if developed with real-scale FTW data, to determine the interaction between FTW and retrofitted lagoons, compartments and constitute a tool for predicting FTW potential treatment benefits.

From an operation point of view, the FTW management strategy should address plant harvesting and sediment dredging frequency to avoid pollutant return to the system.

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AUTHORS' STATEMENT

Rita Abi Hanna: data collection and curation, formal analysis, visualization, writing the original draft, and editing. Karine E. Borne: conceptualization, methodology, funding acquisition, supervision, validation, reviewing, and editing. Claire Gérente: conceptualization, methodology, funding acquisition, supervision, and validation. Yves Andres: conceptualization, funding acquisition, and supervision.

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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