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RESEARCH ARTICLE

Distribution coefficients of nitrogen pollutants between water and sediment and their environmental risks in Lingang hybrid constructed wetland fed by industrial tailwater, Tianjin, China

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Abstract
Exploring the fate of nitrogen pollutants in constructed wetlands (CWs) fed by industrial tailwater is significant to strengthen its pollution control and promoting the development of CWs in the field of micro-polluted water treatment. In this study, the distribution coefficients and the environmental risks of nitrogen pollutants between water and sediment of the hybrid CW in Tianjin were systematically investigated. From a spatial perspective, the nitrogen pollutants could be removed in this hybrid CW, and subsurface flow wetland played a key role in nitrogen pollutant removal. From a temporal perspective, the concentration of nitrogen pollutants was largely affected by the dissolved oxygen (DO) and temperature. The distribution coefficient of nitrogen pollutants between water and sediment was further clarified, suggesting that NH_4^+-N was more likely to be enriched in sediments due to microbial process. The overall level of pollution in hybrid CW was moderate according to the nutritional pollution index (NPI) analysis. The risk assessment indicated that timely dredging control measures should be considered to maintain the performance of hybrid CW.

Keywords Hybrid constructed wetland · Nitrogen pollutants · Water and sediment · Distribution coefficients · Industrial tailwater

Introduction

Constructed wetlands (CWs) are considered to be the environmentally friendly ecosystems that can perform advanced treatment of micro-polluted water, and they have been widely used in the purification treatment of urban sewage, domestic sewage, and industrial sewage (Wang et al. 2020a; Wang et al. 2021; Zhang et al. 2020). Nitrogen is the important nutrient element that affect the growth of plants, but the excessive amounts of nitrogen in water would cause eutrophication and threaten water ecology (Saeed and Sun 2012). Industrial tailwater contains an amount of nitrogen pollutants; thus, it is of great significance to adopt CWs for advanced treatment before industrial tailwater are directly discharged.

CWs are generally divided into surface flow constructed wetlands, horizontal subsurface flow constructed wetlands, and vertical flow constructed wetlands. Njenga et al. (2013) proposed that the surface flow wetland has the advantages of low investment and simple operation, but it is easily affected by the season and hydraulic load; other researchers proposed a horizontal subsurface flow constructed wetland (SF CW) that can handle high pollution loads but is limited by the content of DO (Ferreira et al. 2020; Wang et al. 2020c); vertical flow constructed wetland (VF CW) is also used, which can perform both aerobic nitrification and anaerobic denitrification but is often limited by the influence of intermittent water inflow (Nawaz et al. 2019). Compared with traditional single CW, hybrid CW has significant advantages in the

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efficient treatment of high-load industrial tailwater (Chen et al. 2021; Zhang et al. 2020; Zhu et al. 2021). In this regard, exploring the occurrence of nitrogen pollutants in the hybrid CW is of great significance for optimizing the construction of the hybrid CW.

Previous studies have shown that there are obvious differences in the distribution of nitrogen pollutants between water and sediment (Song 2010). When the water body has serious pollution, part of the exchangeable nitrogen can enter the sediments through precipitation, adsorption, etc., causing serious external source pollution problems (Liu et al. 2016). Even if the external pollution is effectively controlled, the fixed ammonium in the sediment will still be released to the overlying water body, causing internal pollution (Eila et al. 2003; Hong et al. 2019). Moreover, the spatiotemporal distribution of nitrogen pollutants between water and sediment in CWs is usually affected by microbes and physical and chemical properties (Wang et al. 2020b; Zheng et al. 2016). For instance, nitrification of microorganisms is the main factor affecting the distribution variations of NO_3^--N and NH_4^+-N in water, and the microorganisms in sediments mainly convert organic nitrogen into NH_4^+-N through ammonia oxidation (Zhang et al. 2014). Moreover, physical and chemical properties such as DO and temperature also affect the distribution of nitrogen pollutants between water and sediment. DO and temperature under appropriate conditions are conducive to the removal and transformation of NH_4^+-N and NO_3^--N (Li et al. 2019; Ma et al. 2021). Therefore, understanding the spatiotemporal distribution of nitrogen pollutants between water and sediment has important guiding significance for environmental risk assessment. However, studies on the occurrence characteristics of nitrogen pollutants between water and sediment in a typical hybrid CW fed by industrial tailwater are limited.

Hence, in this study, Lingang hybrid CW, Tianjin, which fed by the industrial tailwater, was selected as the object. In autumn and winter, sampling and testing the water and sediments of the hybrid CW were done (1) to study the spatiotemporal variation characteristics of nitrogen contaminations between water and sediment of the hybrid CW, (2) to analyze the distribution law of nitrogen pollutants between water and sediment, and (3) to assess the environmental risk of nitrogen pollutants in the hybrid CW by using the method of nutrient pollution index (NPI). The work for the understanding of the characteristics and distribution of nitrogen pollutants between water and sediment to strengthen hybrid CW ecosystem management and control is important.

Materials and methods

Study site and sample collection

This site (38° 55′ 58″ N, 117° 41′ 58″ E) is a hybrid constructed wetland ecosystem with the theme of industrial sewage treatment. Except for the water inlet and outlet, hybrid CW was roughly divided into four functional areas: regulation pond, subsurface flow wetland (SSF CW), SF CW, and landscape lake. The hybrid CW is about 6.3×10^5 m^2, of which the water accounts for about 1.7×10^5 m^2. In addition, the industrial tailwater of the Shengke wastewater treatment plant is the source of water in hybrid CW.

The study began in the autumn and winter seasons of 2019 (from August to December), and samples were collected on the first 3 days of each month. According to the hydrological characteristics of the hybrid CW, a total of 9 sampling points has been set up (Fig. 1). The collected water samples were put into polyethylene bottles, brought back and stored into the refrigerator at 4 ℃. The collected sediment samples were put into sealed bags and stored into the refrigerator at −20 ℃. All collected samples were tested within 1 week.

Analysis methods

In this study, the nitrogen pollutants included TN, NH_4^+-N, NO_3^--N, and NO_2^--N. At the same time, DO and temperature will also affect the conversion of nitrogen pollutants. The methodology used to measure the above indicators is described in our previous work (Li et al. 2021). The concentration of TN and NO_2^--N was determined by UV spectrophotometry. The concentration of NH_4^+-N was determined by measuring the absorbance value at the wavelength of 420 nm using Nessler's reagent. The concentration of NO_3^--N was determined by measuring the absorbance at 540 nm using N-(1-naphthalene)-diaminoethane photometry method. In addition, a portable multi-parameter measuring instrument (Multi 3630 IDS, WTW, Germany) was used to measure DO and temperature at the sampling point. Data were analyzed by Origin 2017; the relative deviation is within 5%.

Quality assurance and quality control

This research strictly complied with the environmental quality standards and adopted blank, repeated, and standard reference analysis for nitrogen pollutants. This research followed quality assurance and quality control procedures during sampling and analysis. Briefly, Milli-Q water was used to clean the outside of the bottle. Glass and polypropylene vessels were soaked in 10% HNO_3 of 48 h and rinsed with deionized water 3 times after being applied. Each reagent and standard solution were

Fig. 1 CW sampling point layout. **1** From water inlet; **2** from regulation pond; **3–4** from subsurface flow wetland (SSF CW); **5** from surface flow wetland (SF CW); **6–8** from landscape lake; **9** water outlet

guaranteed levels. Each sample was carried out in 3 replicates, and the mean as the final value.

Results and discussion

Spatiotemporal variations of nitrogen

Spatiotemporal variations of nitrogen in water

In water, the spatial variation of nitrogen pollutants was shown (Fig. 2). As shown in Fig. 2a, the concentration of NO_3^--N was between 0.31 and 1.46 mg/L. On the whole, the concentration of NO_3^--N falls and then rises along the flow direction. In the regulation pond, the NO_3^--N concentration dropped by 0.13 mg/L, which may be caused by the adsorption of sediment in the regulation pond (Tunçsiper 2020). Along the water flow direction, NO_3^--N flowed from the regulation pond to the SSF CW and SF CW, respectively. In the SSF CW, the concentration of NO_3^--N changed greatly, and the other study has reported that due to the presence of numerous microbial community (Zhao et al. 2021), NH_4^+-N could be easily converted into NO_3^--N by nitrification. Meanwhile, the NO_3^--N concentration decreased to 0.36 mg/L in the SF CW. We speculated that this may be related to the absorption of plants such as reeds. From the landscape lakes to the water outlet, the NO_3^--N concentration increased slightly from 0.31 to 0.42 mg/L. This is attributed to the microorganism that produced part of NO_3^--N through nitrification (Li et al. 2019).

Similarly, the spatial changes of NH_4^+-N were shown in Fig. 2b. Overall, the concentration of NH_4^+-N between 0.15 and 0.6 mg/L decreased and then increased with the direction of the flow. Compared with other functional areas, the content of NH_4^+-N in SSF CW was relatively low (0.15 mg/L). Our obtained results were consistent with previous research, which assumed that nitrification by microorganisms and adsorption by substrates will occur in SSF CW (Langergraber et al. 2009; Zhao et al. 2021). The spatial variation of NO_2^--N in hybrid CW was shown in Fig. 2c. It was worth mentioning that the concentration of nitrate nitrogen was relatively high at the inlet. In the purification

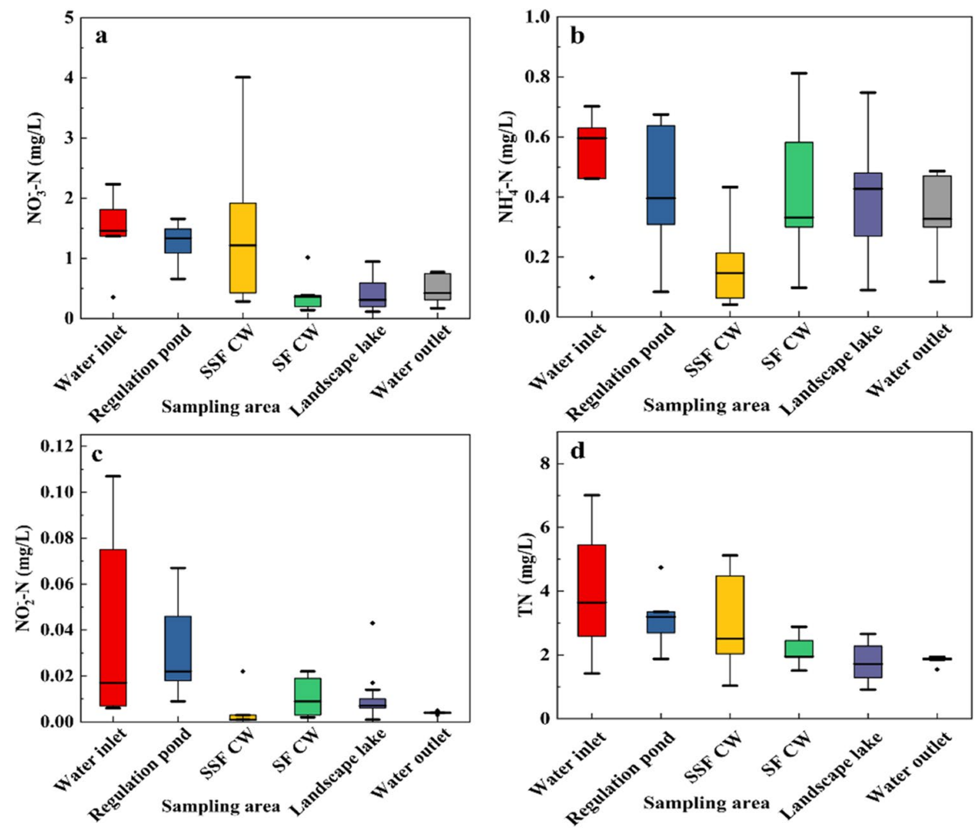


Fig. 2 Spatial variation of **a** NO_3^--N, **b** NH_4^+-N, **c** NO_3^--N, **d** TN in water

of wetlands, the concentration at the water outlet was almost negligible. The maximum concentration of NO_2^--N was not more than 0.022 mg/L in hybrid CW, which could be due to the fact that NO_2^--N was the intermediate product of nitrification and was easily to be converted (Li et al. 2018). However, the NO_2^--N concentration was relatively high at the water inlet, which may be caused by the discharge from the sewage treatment plant.

As shown in Fig. 2d, the concentration of TN ranged from 1.72 to 3.64 mg/L, which indicated a downward trend overall and slightly increased at the outlet. It could be inferred from Fig. 2 that NO_3^--N has a large proportion in TN and was the most important form of nitrogen.

Correspondingly, Fig. 3 showed that the temporal variation of nitrogen from August to December in hybrid CW. From August to December, the temperature decreased month by month, and the content of DO oxygen showed a rising change. The average temperature dropped from 27.8 to 11.4 °C (Fig. S1), and the DO content increased gradually from 8.2 to 10.5 mg/L (Fig. S2). Overall, the concentration of NO_3^--N first decreased and then increased with time (Fig. 3a). As the temperature decreased slowly, the conversion of NO_3^--N was not affected, causing the content of NO_3^--N dropped from 1.02 to 0.28 mg/L before October. Next, it gradually increased to 0.74 mg/L in December, which may be due to the secondary release of a few uncleared plants. Meanwhile, when the temperature was lower than 15 ℃ in December (Fig. S1), nitrification and denitrification could not be carried out effectively, leading to the slightly accumulated of NO_3^--N.

Analogously, Fig. 3b showed the temporal variation of NH_4^+-N. The NH_4^+-N concentration showed an overall upward trend with time (from 0.10 to 0.63 mg/L), which could be due to the slowing down of nitrification and the death of organisms as the temperature gradually decreases (Ding et al. 2017; Man et al. 2022). As shown in Fig. 3c, the concentration of NO_2^--N changed with time. Interestingly, the fluctuation range of NO_2^--N content was smaller in time. Hence, it could be deduced that the temporal and spatial changes of NO_2^--N were consistent.

The temporal variation of TN concentration was shown in Fig. 3d. From August to September, it showed an upward trend from 2.20 to 2. 66 mg/L. The variation trend of TN concentration from September to December was similar to NO_3^--N. The TN level showed a downward trend from 2.66 to 1.70 mg/L and then gradually increased to 2.05 mg/L in December. It can be concluded from Fig. 3 that NO_3^--N accounted for the highest proportion of TN in the temporal variation, which was also reported in previous studies (Li et al. 2015).

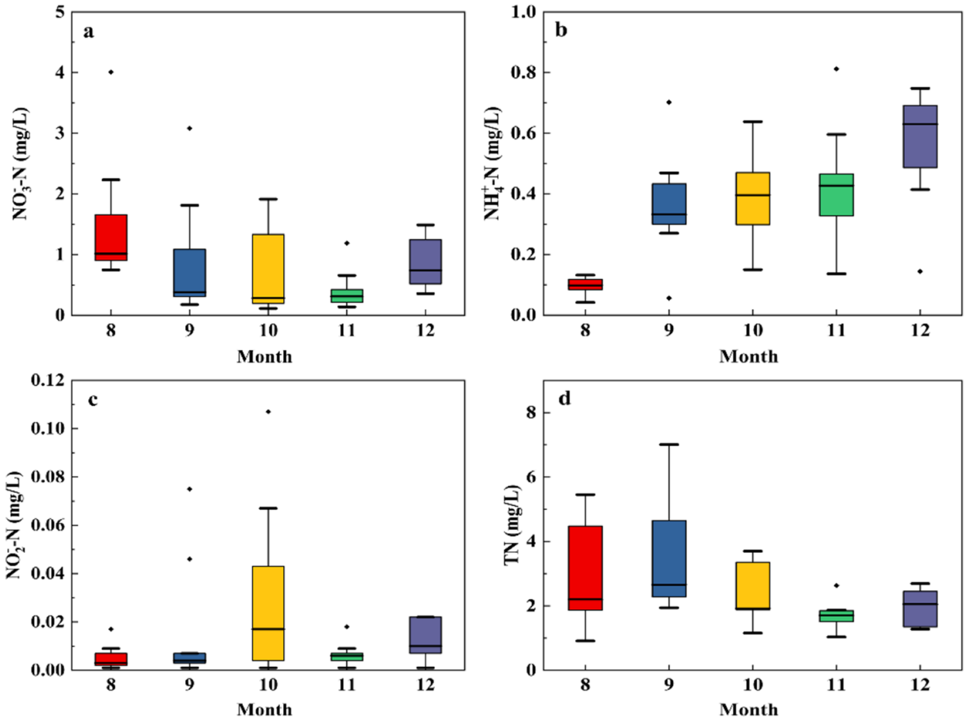


Fig. 3 Temporal variation of **a** NO_3^--N, **b** NH_4^+-N, **c** NO_2^--N, and **d** TN in water

Spatiotemporal variation of nitrogen in sediments

The spatial variation of nitrogen pollutant concentration in sediments of hybrid CW was shown in Fig. 4. As shown in Fig. 4a, the content of NO_3^--N in sediments flowed from the water inlet to the SSF CW and the SF CW, and all of them showed a slight downward from 2.37 to 1.19 and 1.37 mg/kg, respectively. This may be attributed to the substrate adsorption of in the SF CW and the plants absorption in the SSF CW (Saeed and Sun 2012). In the landscape lake, the NO_3^--N concentration decreased to 0.72 mg/kg, which may be due to the low DO in the landscape lake sediments (Fig. S2). At the same time, in the hypoxic environment, the nitrification of microorganisms slows down, and it is conducive to the progress of denitrification (Hong et al. 2019). After that, it showed a slight upward trend at the water outlet (up to 1.32 mg/kg), which may be due to the flow rate of the water outlet was slow and the sediment fully adsorbed NO_3^--N in water.

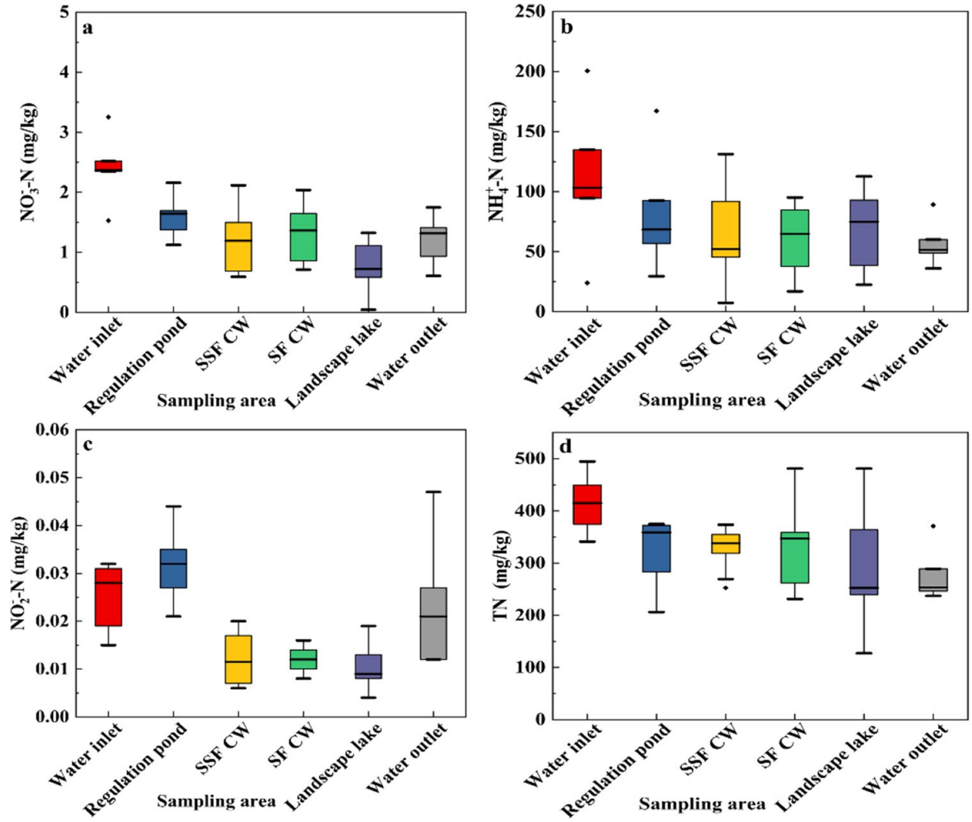
The spatial variation of NH_4^+-N in sediments was shown in Fig. 4b. At the water inlet, the NH_4^+-N concentration was 103.28 mg/kg. The NH_4^+-N concentration from the regulation pond to the water outlet was 51.52 to 68.48 mg/kg. Except for the water inlet, the NH_4^+-N concentration in other regions showed little difference. However, it was obviously higher in sediments than that in water, which was mainly attributed to two aspects: on the one hand, the adsorption of the matrix leads to the migration of NH_4^+-N in water to sediments (Wu et al. 2018); on the other hand, the NH_4^+-N in sediments mainly came from the mineralization process of total organic nitrogen (TON) (Zhu et al. 2019). And the low DO slows down nitrification, which leads to an upward trend of NH_4^+-N accumulation in the sediments. According to Fig. 4c, the concentration of NO_2^--N was extremely low due to poor stability (range from 0.009 to 0.032 mg/kg).

The spatial variation of TN in sediments was shown in Fig. 4d. The variation trend of TN concentration in sediments was similar to NH_4^+-N. The TN levels ranged from 252.40 to 414.84 mg/kg. TN at the water inlet was high (414.84 mg/kg) and then decreased to 358.76 mg/kg in the regulation pond and with little change in the SSF CW and SF CW (337.88 and 347.00 mg/kg respectively). It decreased to 252.40 mg/kg in the landscape lake and finally recovered slightly to 253.60 mg/kg.

The temporal variation of NO_3^--N in sediments was shown in Fig. 5a. The NO_3^--N concentration changed in a small range from August to December (ranging from 0.82 to 1.37 mg/kg), which is related to the concentration of DO. As the temperature decreases, the DO of the water shows an upward trend as shown in Fig. S2. On the contrary, low concentration of DO in sediments is a key limiting factor for removal of NO_3^--N.

The NH_4^+-N value fluctuated greatly from August to December. In August, the concentration of NH_4^+-N was only 24.00 mg/kg. This is related to the migration of pollutants in water. By comparison, it is found that the pollutant level in the water was also low. After that, the NH_4^+-N concentration showed an upward trend in September and October

Fig. 4 Spatial variation of **a** NO_3^--N, **b** NH_4^+-N, **c** NO_2^--N, and **d** TN in sediments



(95.12 and 91.76 mg/kg, respectively). This may be due to the increase of NH_4^+-N content in water, resulting in a corresponding increase in sediments, and the mineralization of organic nitrogen also produced part of NH_4^+-N (Zhu et al. 2019). However, the NH_4^+-N concentrations in November and December were 56.80 and 60.16 mg/kg, respectively.

Fig. 5 Temporal distribution of **a** NO_3^--N, **b** NH_4^+-N, **c** NO_2^--N, and **d** TN in sediments

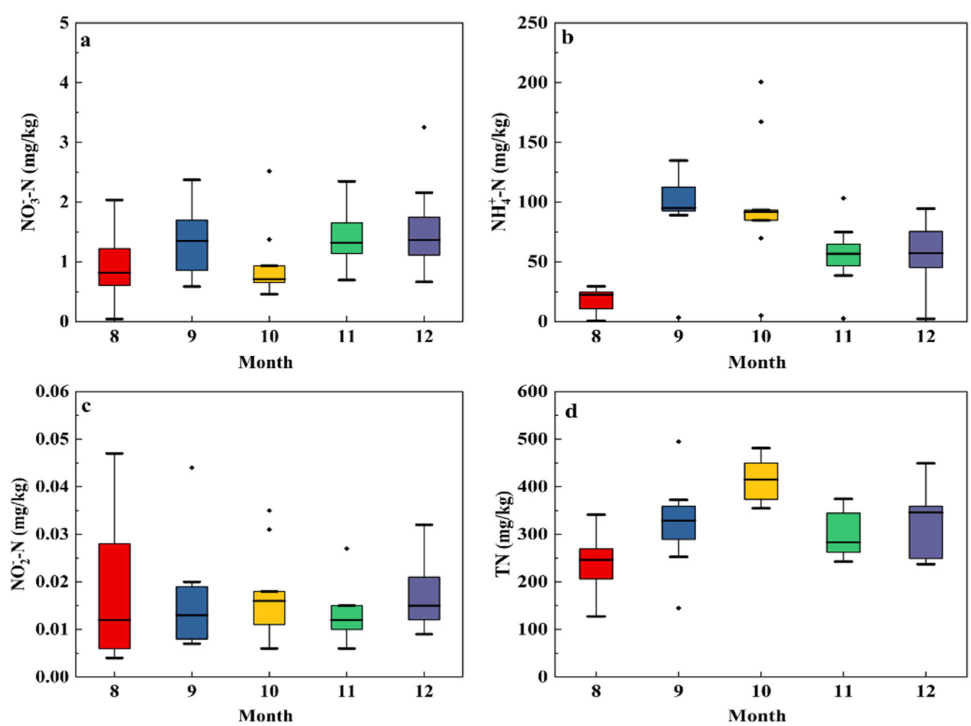


Table 1 Distribution coefficient of different regions of hybrid CW (L/kg)

	Water inlet	Regulation pond	SSF CW	SF CW	Landscape lake	Water outlet	Mean
NO_3^--N	1.63	1.23	0.98	3.81	2.34	3.12	2.19
NH_4^+-N	173.29	172.93	354.29	195.42	175.27	157.55	204.79
NO_2^--N	1.65	1.45	11.5	1.33	1.29	5.25	3.75
TN	113.87	112.25	134.61	177.86	147.00	136.12	136.95

Table 2 Distribution coefficient of hybrid CW in different months (L/kg)

	8	9	10	11	12	Mean
NO_3^--N	0.80	3.54	2.53	4.23	1.84	2.59
NH_4^+-N	244.90	286.51	231.72	133.02	95.49	198.33
NO_2^--N	4.00	3.25	0.94	2.00	1.50	2.34
TN	111.93	123.75	216.06	166.11	168.58	157.29

Since the effect of temperature on microorganism activity and mineralization, NH_4^+-N concentration decreased slightly (Ding et al. 2017). As shown in Fig. 5c, the NO_2^--N concentration remained at a low level (much less than 0.032 mg/kg) and didn't change significantly over time.

The variation trend of TN in sediments from August to December was similar to that of NH_4^+-N. It increased from 246.36 to 414.84 mg/kg (Fig. 5d) and then decreased to 282.88 mg/kg in November and then increased slightly in December (345.92 mg/kg). Therefore, it could be known that NH_4^+-N accounts for the highest proportion of TN, which had been similarly reported in previous studies (Zhu et al. 2019).

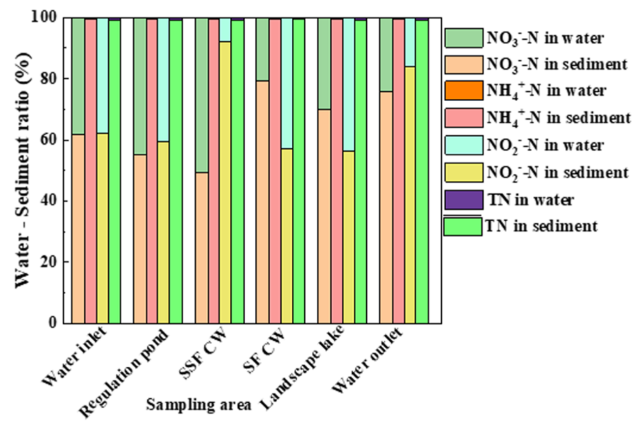


Fig. 6 Spatial distribution of nitrogen pollutants in hybrid CW

Distribution coefficient of nitrogen pollutants between water and sediment

The distribution of nitrogen pollutants between water and sediment was one of the important factors to analyze the rule of migration and transformation. The distribution coefficient (K_d) can be used to evaluate the distribution of pollutants between water and sediment. K_d is calculated according to the concentration ratio between water and sediment (Tang et al. 2019), and its calculation formula is as follows:

$$K_d = \frac{C_s}{C_w} \tag{1}$$

In which C_s and C_w represents the concentration of nitrogen pollutants in sediment and in water, respectively.

The K_d values of N in hybrid CW with the spatiotemporal variation were shown in Tables 1 and 2. As can be seen from Table 1, the average K_d values of NO_3^--N, NH_4^+-N, NO_2^--N, and TN in different areas of hybrid CW were 2.19 L/kg, 204.79 L/kg, 3.75 L/kg, and 136.95 L/kg, respectively. And those from Table 2 were 2.59 L/kg, 198.33 L/kg, 2.34 L/kg, and 157.29 L/kg, respectively. It could be seen that the mean K_d of NH_4^+-N and TN were significantly higher than NO_3^--N and NO_2^--N, in conjunction with Figs. 6 and 7, which indicated that sediments have a strong adsorption effect on NH_4^+-N and TN. From the perspective of spatial distribution, as shown in Fig. 6, the proportion of nitrogen pollution in sediments in different regions was significantly higher than that in water bodies. Similarly, from the perspective of temporal distribution, as shown in Fig. 7, the distribution of nitrogen pollutants was consistent with Fig. 6, which was mainly determined by its distribution coefficient. Therefore, the influence of NH_4^+-N and TN in water was less than that in sediments, and they were more easily enriched in sediments than in water, which parallels previous reports (Zhu et al. 2019). However, the average K_d values of NO_3^--N and NO_2^--N were small, which indicated that both of the adsorption effects between water and sediments were not different.

Pearson correlation analysis of nitrogen pollutants in different areas of CWs was analyzed (Fig. S3). The distribution of NH_4^+-N and TN displayed a good correlation between water and sediment (the correlation coefficients are 0.91

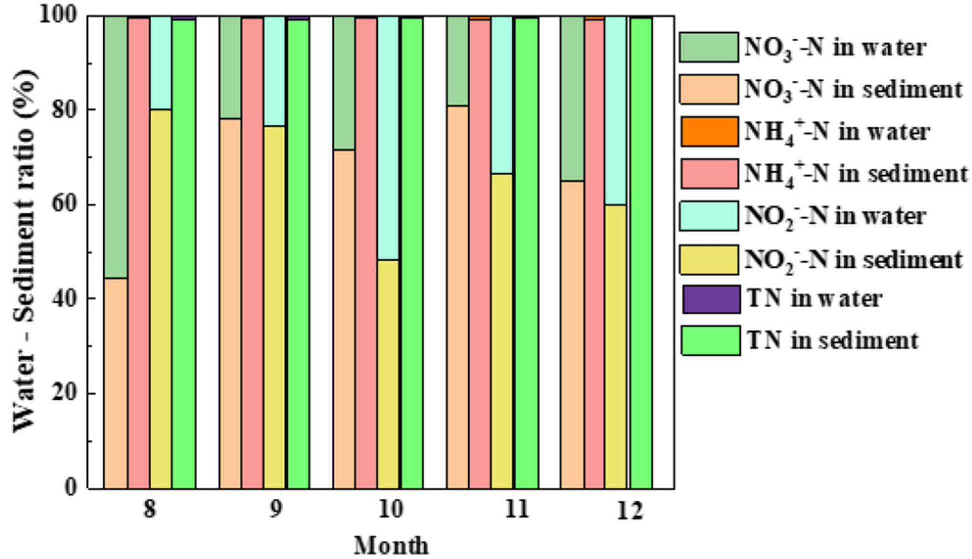


Fig. 7 Temporal distribution of nitrogen pollutants in hybrid CW

Table 3 Nutritional pollution index

	Water inlet	Regulation pond	SSF CW	SF CW	Landscape lake	Water outlet
NPI	3.09	2.46	2.14	1.73	1.47	1.46

and 0.87, respectively). This phenomenon indicated that in autumn and winter, the ammonia nitrogen and total nitrogen in the sediment would be released into the water body again, causing the corresponding concentration of the water body to increase. The correlation between nitrate nitrogen and nitrous acid was not significant, which was in line with the results of Li et al. (2020).

Environmental risk assessment

Nutritional pollution index (NPI) can effectively describe the pollution of nitrogen pollutants to water quality and the environment (Isiuku and Enyoh 2020). In addition to NO_2^--N, the above three forms of nitrogen were important factors causing water eutrophication. Therefore, the pollution analysis of NO_3^--N, NH_4^+-N, and TN was carried out (NO_2^--N was not considered because of its small content in water). The NPI calculation formula was as follows:

$$NPI = \frac{C_1}{MAC_1} + \frac{C_2}{MAC_2} + \frac{C_3}{MAC_3} \quad (2)$$

C_1, C_2, and C_3 represent the average concentration of NO_3^--N, NH_4^+-N, and TN in water, respectively. MAC_1, MAC_2, and MAC_3 are 20, 1.5, and 1.5 mg/L which correspond to the above maximum standard concentrations in turn, respectively. Classification criteria of NPI are as follows: $NPI < 1$ (no pollution), $1 \leq NPI \leq 3$ (moderate pollution), $3 < NPI \leq 6$ (high pollution), and $NPI > 6$ (severe pollution).

The distribution of the nutrition pollution index (NPI) was shown in Table 3 and Fig. 8. As shown in Table 3, it can be found that the NPI in the water inlet was 3.09, which reflects that the pollution degree of industrial tailwater is highly polluted. When the water source flows

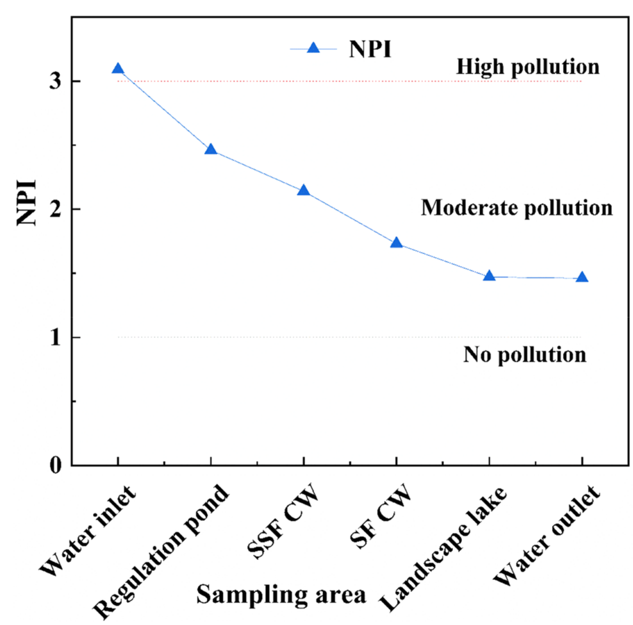


Fig. 8 Nutrient pollution index of different forms of nitrogen

through the regulation pond, the NPI quickly drops to 2.46, and the water quality is significantly improved. In the same way, along the direction of the water flow, the nutrient pollution index gradually decreases to 1.46. With reference to Fig. 8, it is clearly observed that except for the water inlet, the pollution levels of other areas were all moderately polluted, and the water quality at the water outlet is getting closer and closer to the pollution-free level. Interestingly, the water quality changes most obviously in the regulating pond. Through the temporal and spatial distribution of nitrogen pollutants in water and sediment, it can be inferred that NO_3^--N in the water was the main component that causes the moderate pollution of the hybrid CW. Moreover, sediments containing large amounts of NH_4^+-N and TN are at risk of secondary release. Therefore, it is recommended to take preventive measures to dredge in time.

Conclusion

In this study, the spatiotemporal variation characteristics of nitrogen in water and sediment of hybrid CW were analyzed, and the environmental risk was studied. The results showed that the main nitrogen form in water was NO_3^--N (0.31–1.46 mg/L), while NH_4^+-N (51.52–103.28 mg/L) was the main nitrogen form in sediments. The distribution coefficient showed that it has a strong adsorption effect on NH_4^+-N and TN, and both of which were more easily enriched in the sediment. By comparing the experimental results, the concentration of nitrogen pollutants had a positive correlation between water and sediments. In addition, the nutrition pollution index (NPI) showed that the wetland as a whole was at a moderate pollution level, and NO_3^--N was the important factor affecting the water pollution of hybrid CW.

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Author contribution Xiaodong Ma, Gengbo Ren, and Hongrui Li conceived and designed the study. Hongrui Li and Peng Gao performed experiments. Quanli Man and Hongrui Li wrote the manuscript. Xiaodong Ma, Gengbo Ren, Bin Zhou, and Honglei Liu revised the manuscript. All authors read and approved the final manuscript.

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Data availability All relevant data generated or analyzed during this study were included in this published article.

Declarations

Ethics approval and consent to participate This manuscript does not contain any studies with human participants or animals performed by any of the authors.

Consent for publication All authors give consent to publish the research in Environmental Science and Pollution Research.

Competing interests The authors declare no competing interests.

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