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Influencing Factors and Nutrient Release from Sediments in the Water Level Fluctuation Zone of Biliuhe Reservoir, a Drinking Water Reservoir

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Abstract: Significant amounts of nitrogen and phosphorus in sediments will be released into the overlying water during the flood season in the water level fluctuation zone (WLFZ) of reservoirs that undergo periodic drying and flooding. This will result in water quality deterioration of the reservoir. In order to clarify the distribution characteristics and release behavior of nitrogen (N) and phosphorus (P) from sediments in the WLFZ of a reservoir, this study analyzed the sediment distribution characteristics and potential exchange flux sediment–water interface(SWI) through field investigations and sediment core incubation experiments. And the main factors affecting the release of N and P through the incubation experiments in sediments of the WLFZ in the reservoir were determined. Our findings indicated that the sediment in the WLFZ serves as the primary source of NH_4^+-N and acts as a sink for NO_2-N in the overlying water of sediment. The concentration of NH_4^+-N in the interstitial water of sediments is the key factor that affects the water quality of Biliuhe Reservoir. Total nitrogen content of surface sediments in the WLFZ of Biliuhe Reservoir ranges from 1052.52 ± 49.39 to 3520.54 ± 30.31 mg/kg. High concentrations of N pollution are the primary increased risk of eutrophication in Biliuhe Reservoir during summer. The sediment N and P release flux of BLH1 located in the main stream is 1.67 ± 1.06 and 12.32 ± 2.42 mg·(m^2·d)$^{-1}$, respectively, which is smaller than that of BLH2 (3.27 ± 2.15 and 15.19 ± 2.36 mg·(m^2·d)$^{-1}$, respectively), BLH3 (4.24 ± 1.74 and 17.02 ± 2.47 mg·(m^2·d)$^{-1}$, respectively) and BLH4 (7.78 ± 2.03 and 20.56 ± 2.38 mg·(m^2·d)$^{-1}$, respectively) located in the tributary. It indicates that the water conveyance project located in BLH1 has an impact on nutrient scouring of sediments in the WLFZ at this site. The main water environment factor affecting the release of N and P in the sediment of the WLFZ is dissolved oxygen (DO). And the Pearson correlation coefficients between TN and TP with DO were −0.838 and −0.777, respectively ($p < 0.05$). At the same time, the diffusion of nutrients in the sediments can be effectively inhibited by maintaining a certain DO concentration in the overlying water.

Keywords: nitrogen; phosphorus; release; sediment; water level fluctuation zone; Biliuhe Reservoir

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Citation: Li, W.; Xu, S.; Chen, X.; Han, D.; Mu, B. Influencing Factors and Nutrient Release from Sediments in the Water Level Fluctuation Zone of Biliuhe Reservoir, a Drinking Water Reservoir. *Water* **2023**, *15*, 3659. https://doi.org/10.3390/w15203659

Academic Editor: Catherine N. Mulligan

Received: 17 September 2023
Revised: 9 October 2023
Accepted: 17 October 2023
Published: 19 October 2023

1. Introduction

The water level fluctuation zone in a reservoir refers to an area where the submerged region undergoes periodic drying and flooding due to operational management, also known as the fluctuation zone. When the WLFZ is dried and then covered with water, a large amount of nutrients that have accumulated during the drying period of the sediment is released into the overlying water. These nutrients mainly include phosphorus and nitrogen. In terms of P, its forms are relatively stable and easily released from the sediment into the overlying water. As for N, under aerobic conditions, ammonium is transformed into nitrate by nitrifying bacteria. When the dissolved oxygen (DO) concentration is low in the aquatic system, the denitrification process becomes dominant. At this time, nitrite is reduced to N_2 and removed from the sediment–water system [1]. This portion of the water area constitutes a significant proportion of the reservoir's total area and persists for an extended period [2]. Simultaneously, the water is susceptible to wind and wave disturbances, which result in the resuspension of sediment and release of nutrients into the overlying water, thereby compromising its quality [3]. Additionally, this region is inferior to deeper waters due to the high risk for nutrient release from sediments and subsequent algal blooms. The WLFZ of the reservoir experiences strong light exposure, leading to higher temperatures and oxidation that reduces DO concentration in the overlying water within this area. Therefore, the WLFZ of the reservoir, affected by alternating flooding and drying, is primarily influenced by specific factors such as light, temperature, DO and wind–wave disturbances. These factors enhance the likelihood of nutrient release from the sediment to the overlying water, which can cause a change in the environment and deteriorate reservoir water quality due to runoff input. Previous studies have demonstrated that a substantial proportion of lakes and reservoirs worldwide, ranging from 30% to 55%, exhibit severe eutrophication, with freshwater eutrophication in the United States alone resulting in potential economic losses exceeding USD 200 million [4]. Therefore, protecting and treating this zone is crucial for maintaining overall reservoir water quality.

Under the influence of periodic fluctuations in reservoir water level, the WLFZ has experienced different degrees of the drying–flooding cycle process. During the drying phase, aerobic conditions facilitate rapid microbial proliferation, resulting in P enrichment within the burgeoning biological community. As moisture levels decrease and microorganisms die off, organic matter decomposition and mineralization are stimulated by the drying [5], resulting in active P accumulation at the surface layer. When the soil is flooded, a significant amount of ammonia nitrogen (NH_4^+-N), nitrate nitrogen (NO_3-N) and total nitrogen (TN) are released into the overlying water. This release increases by five times, resulting in the discharge of accumulated N and P during sediment exposure and drying periods. High phosphorus concentration in old sediment may contribute to dense phytoplankton blooms by supplying phosphorus to the water [6]. Consequently, this elevates eutrophication risks within the reservoir area. Previous studies have demonstrated that the sediments in the WLFZ are influenced by the drying–flooding cycle, which in turn affects geochemical processes such as element circulation and transformation, material release and interception, pollutant purification, and sediment transport in the aquatic ecosystem [7,8]. When exogenous pollution is gradually, effectively controlled, the key to the treatment of lake and reservoir water becomes how to effectively control the endogenous N and P load from the sediment of the WLFZ [9]. The risk of N and P release is most closely related to the form of N and P, the characteristics of the overlying water environment, the characteristics of the N and P profiles in sediment interstitial water, porosity and the organic matter concentration. Therefore, it is of great significance to investigate the vertical distribution of N and P in sediment, interstitial water, and overlying water as well as to identify the migration flux and factors associated with sediment release. This will contribute to studying the eutrophication status and analyzing pollution sources in reservoirs.

Currently, the research on the deposition release behavior of nutrients focused on the coastal, tidal flat areas, as well as the WLFZs of lakes and reservoirs [10–13]. Brödlin et al. [14] have demonstrated that alternating periods of flooding and drying can enhance the release of endogenous nutrients in sediments with continuous water cover. Under aerobic conditions during the initial exposure stage, rapid microbial growth led to P enrichment in the developing biological community. When further drying, microorganisms died as the water content decreased. Garcia thinks that drying could promote the decomposition and mineralization of organic matter [5]. When the soil is flooded again, a large amount of NH_4^+-N, NO_3-N and TN are released into the overlying water, which increases the risk of eutrophication in the reservoir area. The release of N and P from sediments to the overlying water is not only influenced by the physical and chemical properties of the flooded soil, but it is also impacted by internal factors (such as the forms of N and P in overlying water and sediments) and external factors (such as water environment factors) [15]. Specific external factors include overlying water nutrient concentration, dissolved oxygen (DO), pH, oxidation-reduction potential (ORP), biomass of algae in overlying water, microbial activity, interference from water, intensity of external inputs, interface temperature, etc. [16,17]. Internal factors include sediment interstitial water nutrient concentration, content and forms of N and P in sediment, sediment grain size, content and forms of metal ions, pH and ORP. Other scholars argue that sediment activity and organic matter content are the primary influencing factors of N diffusion flux. The content and form of P in sediments, on the other hand, significantly affects the P diffusion flux of the sediment [18]. The concentration of DO is also the main factor affecting the release of N and P. The laboratory static simulations showed that anaerobic conditions with DO less than 0.5 mg/L accelerated the release of P from the sediment, while aerobic conditions with DO greater than 5.0 mg/L inhibited the release of P from the sediment [19]. In general, at high DO levels, the water exhibits an aerobic state, which inhibits denitrification in sediments and reduces NO_3-N consumption. Simultaneously, aerobic conditions can inhibit the dissimilatory reduction of NO_3-N to NH_4^+-N, promote nitrification, help prevent sediment release, and limit the diffusion of PO_4^{3-} in interstitial water into overlying water. Conversely, anaerobic conditions will accelerate the release of pollutants in sediments. In the environment where algae grow vigorously, the explosive growth of algae consumes a large amount of available N and P in water, and P supply capacity of sediments tends to be passively exerted to a higher or highest level, prompting P to migrate to the sediment [20]. Among these environmental factors, temperature is a key determinant that controls the release of endogenous N and P in aquatic ecosystems [21]. Studies have shown that an increase in water temperature will lead to an increase in the release rate of N and P and the release amount of P [22].

This study creatively employed a combination of field investigation and indoor incubation experiments to reveal the underlying regularity and mechanism governing the release of N and P nutrients into the overlying water when dry sediments in the WLFZ of the reservoir are submerged. The drinking water reservoir is an essential source for urban drinking water supply [23], which has a higher requirement for water quality and is closely related to the safety of citizens' drinking water [24]. Biliuhe Reservoir, located in Liaoning Province, China, serves as the main urban water supply source of Dalian City. A total of 80% of Dalian's water supply comes from Biliuhe Reservoir, with a daily water supply reaching 1.2 million m^3. Therefore, the sediment and overlying water at the flooding and drying alternation zone of the WLFZ were selected for investigation in Biliuhe Reservoir as it is an important and special area in the water environment. The objective of this study is to investigate the characteristics of N and P sources and sinks in the sediment of the WLFZ, as well as the regularity and mechanisms governing N and P deposition. The temporal variation characteristics of water environmental factors in each water column were analyzed during the migration of N and P in sediment within the WLFZ of the reservoir, along with the corresponding response relationship between overlying water environmental factors and N/P release from the sediment to identify the water environmental factors that affect the migration flux of N and P in the sediment of the WLFZ. The results of this study are expected to elucidate the deposition release mechanism of N and P in the WLFZ, providing a reference for controlling endogenous nutrient release independent of other environmental factors.

2. Materials and Methods

2.1. Site Description

In this study, the WLFZ of Biliuhe Reservoir was selected as the research area. Biliuhe Reservoir is located at Liaoning Province, China (as shown in Figure 1). The reservoir has a total storage capacity of 934 million m^3, with a normal high water level of 69 m, a flood limit water level of 68.1 m, and a minimum water level of 47.0 m. Additionally, the control area above the dam site covers an area of 2085 square kilometers. Biliuhe Reservoir supplies 80% of Dalian's water, and the daily water supply reaches 1.2 million m^3. In addition to the main stream of Biliu River, there are three main tributaries, namely Geli River, Bajia River and Yaba ditch. There are also several small tributaries that serve as important water sources for domestic drinking water and industrial and agricultural use in Dalian City. Furthermore, a water diversion project has been implemented to introduce Dahuofang Reservoir's water into Biliuhe Reservoir.

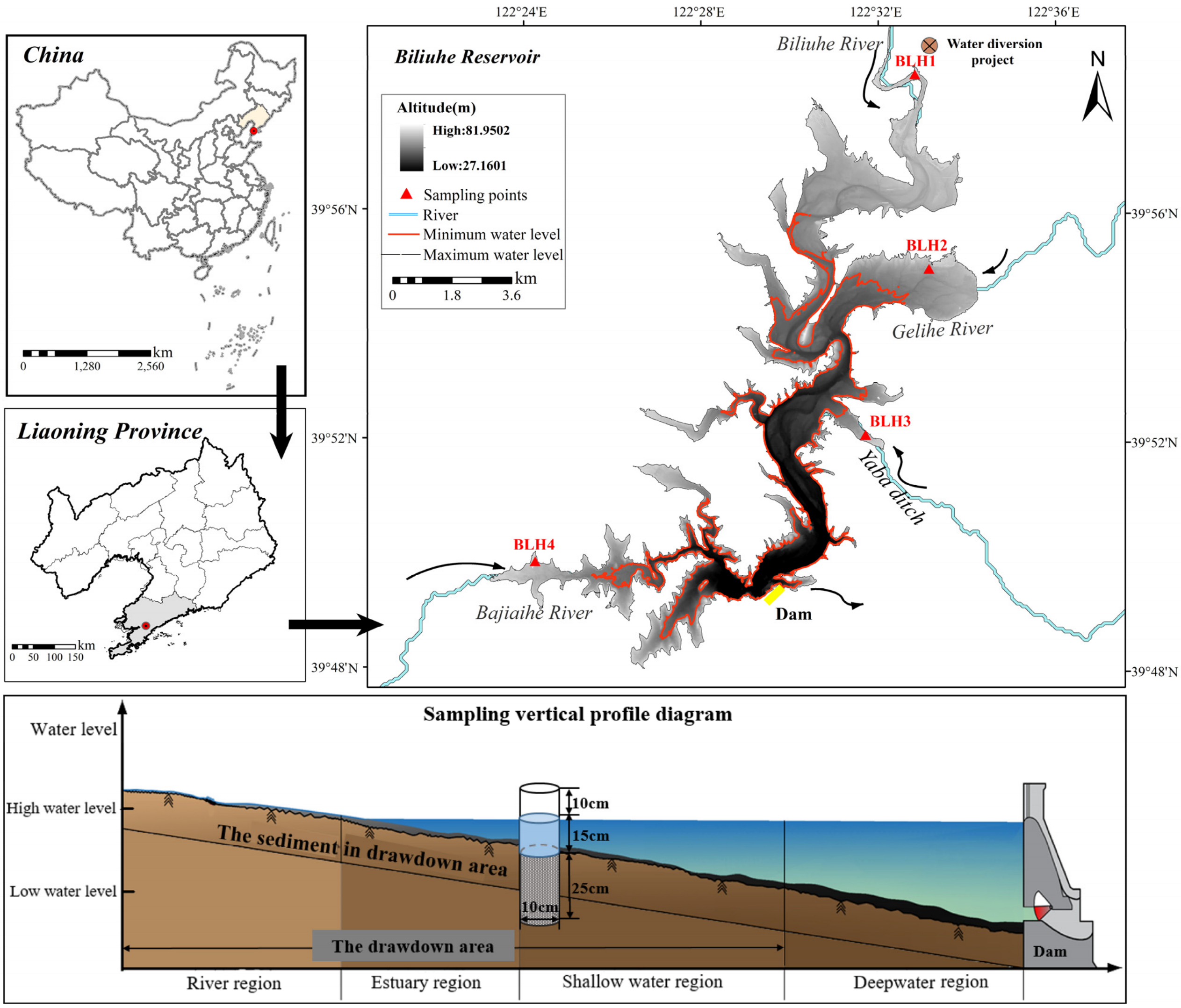
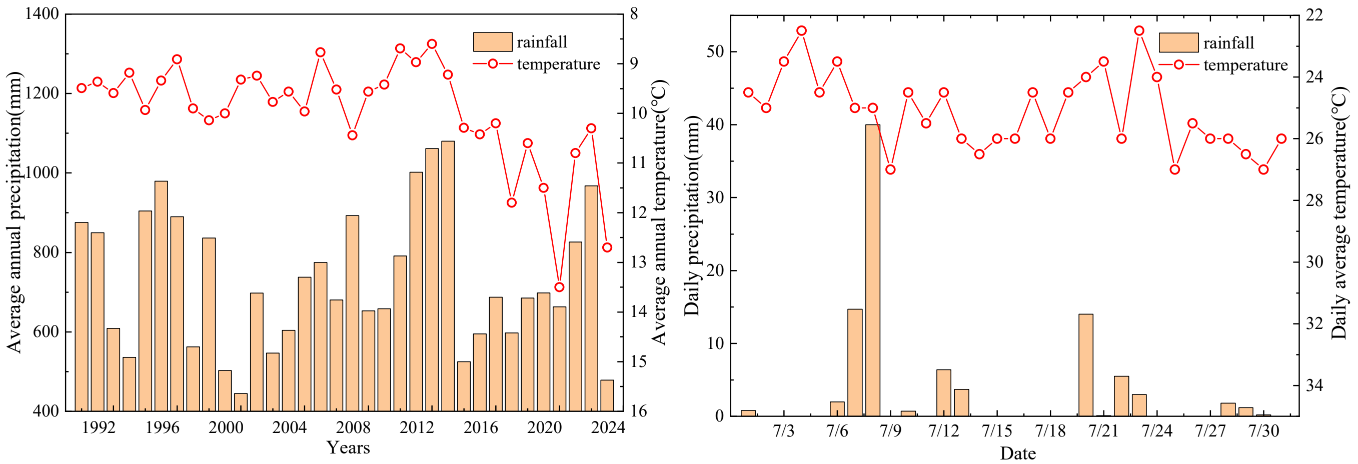


Figure 1. Location of Biliuhe Reservoir and distribution of sampling sites.

The annual average area of the three tributaries was 28.709 km^2 during the dry period in the WLFZ. The temporal behavior of the precipitation and temperature of Biliuhe Reservoir were analyzed. The average annual precipitation in the basin is 746 mm, and the average annual temperature is 10.6 °C (Figure 2). It belongs to the temperate oceanic monsoon climate and is a typical river-type reservoir. The annual variation in precipitation is significant. The interannual temperature exhibited an overall declining trend ($p < 0.05$). The observed temperature range in July 2022 ranged from 22.5 °C to 27.4 °C. The average daily temperature in July was 25.11 ± 1.26 °C. The accumulative amount of precipitation in July was 92.1 mm. After the sample collection, a cumulative precipitation of 54.7 mm was recorded in the following two days, which is likely to result in an elevation of the reservoir water level. Consequently, the previously exposed dry sediment will be submerged under water, thereby triggering the release of nutrients from the sediment into the overlying water.

The reservoir has been in operation for over 30 years, and the water quality is good except for N [25]. However, during periods of low water levels, a wide range of the WLFZ is exposed for agricultural planting and plant growth according to the reservoir's operational mode. Despite controlling most point source pollution in recent years, P levels continue to increase.



(**a**) (**b**)

Figure 2. Temporal behavior of temperature and precipitation patterns in the study area. (**a**) The annual average temperature and annual average precipitation of Biliuhe Reservoir over the past 30 years. (**b**) Average daily temperature and daily precipitation for July 2020.

From 1985 to 2022, the water level of Biliuhe Reservoir fluctuates greatly due to the uneven inflow of water from the WLFZ. Under the condition of periodic fluctuations in reservoir water level, the WLFZ is wide, and the drying period is long. The water level of sediment samples collected in the WLFZ of the reservoir measures 66.45 m. Throughout the entire operational period of the reservoir (from 1985 to 2022), there has been a 16.23% probability of submergence frequency at this level (Figure 3), and this area experiences frequent flooding and drying alternations throughout the year.

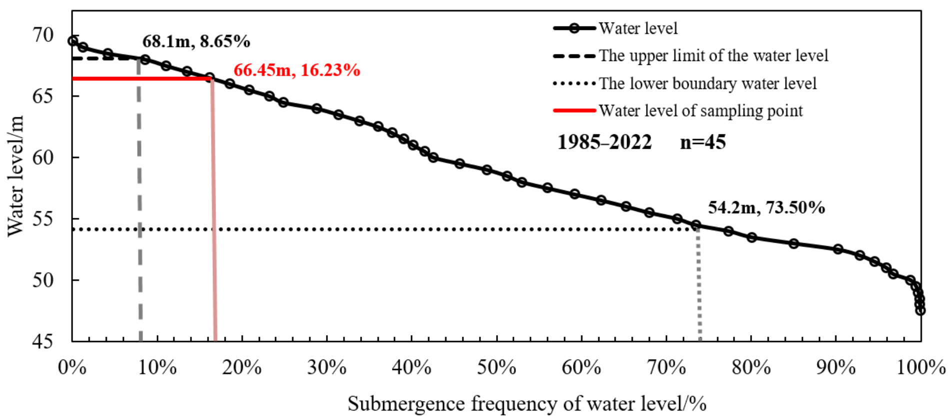


Figure 3. Water level submerged distribution curve.

2.2. Field Sampling

Throughout the entire operational period of the reservoir, there has been a 16.23% probability of submergence frequency at this level. The sediment at this elevation is exposed for an extended duration. As the water level increases, previously exposed sediment nutrients will be released into the overlying water, leading to a deterioration in reservoir water quality.

In order to analyze the regularity of nutrient release in sediments of the WLFZ, the original columnar sediments and overlying water samples were collected in July 2022. The distribution of sampling sites is shown in Figure 1. The latitude and longitude of sampling points are positioned by GPS. Four typical sampling sites (BLH1, BLH2, BLH3 and BLH4) were set up in the main tributaries from upstream to downstream (Figure 1). BLH1 is located in the main stream of the reservoir, where a water diversion project has been implemented to introduce water from Dahuofang Reservoir into Biliuhe Reservoir, resulting in erosion of BLH1 sediment. Meanwhile, BLH2, BLH3 and BLH4 are located in the tributaries of the reservoir. Through field investigation, it was found that BLH2 is located in Geli River, which is located in the agricultural planting area. Both BLH3 and BLH4 are located in residential areas and agricultural planting areas. BLH4, a submerged dam project, was established. It can be seen from Table S1 that the porosity of the sediment core of Biliuhe Reservoir varies from 18.62% to 50.52%. From the upstream to the downstream of the reservoir, the porosity of the sediment core gradually increases.

Columnar sediments were collected vertically with PVC tubes (diameter of 100 mm) in BLH1, BLH2, BLH3 and BLH4. The sampling frequency was five times a month, which implies that a total of five sample collections were conducted in the month of July. Four sampling sites were collected in each sampling, and three parallel experiments were conducted for each sampling site to ensure experimental accuracy. Consequently, a total of 12 columnar sediment experiments were performed to minimize potential errors at each sampling. Finally, the average value of July sampling was taken as the final result. The vertical sampling depth was about 25 cm. This depth was chosen as it falls within the range of water level fluctuations observed in Biliuhe Reservoir over a 20-year period and aligns with global change projections for the 21st century. After taking the columnar sediment sample with a PVC pipe at the site, we quickly used a stopper to block the other end of the pipe to prevent any potential leakage from the columnar sediment. Subsequently, the columnar sediments were sectioned and divided into 5 cm intervals. Finally, the separated 5 cm columnar sediment samples were promptly loaded into a clean polyethylene bag. On the same day, the sediment interstitial water was taken back to the laboratory and extracted by the centrifuge. The columnar samples were collected in a vertical position, without any shaking and with swift sampling, to eliminate the influence of other interlayers of the water. This approach effectively minimizes the impact caused by other interlayers of the water.

After sampling, the collected original column samples were sealed with rubber plugs to prevent the disturbance during transportation from affecting the experimental results. At the same time, the sediments were collected in situ by layers, and a sample was collected every 5 cm depth and placed in a clean polyethylene bag for sealing and low temperature preservation. The samples were transported back to the laboratory and centrifuged to obtain the interstitial water required for the experiment. A YSI EXO2 multiparameter probe (XYLEM, Washington, DC, USA) was used to measure water environmental parameters: pH, water temperature (WT), dissolved oxygen (DO), electrical conductivity (EC), turbidity and chlorophyll-a on the vertical profile of each sampling site. At the same time, 10 L in situ bottom water samples were collected for static release incubation nitrogen and phosphorus experiments. And a Niskin water sampler produced by HYDRO-BIOS, Kiel, Germany was used to collect the overlying water. All water samples collected were transported to the refrigerator within the same day at 4 °C for testing.

2.3. Incubation of Sediment Cores

To study the potential exchange of nutrients at the SWI, three replicates of sediment cores from each site were incubated. The collected sediment samples were transported back to the laboratory, and the overlying water was drained using a siphon method for release experiments. The filtered in situ bottom water sample was slowly and undisturbedly injected into the in situ sediment column in the WLFZ along the wall with a medical infusion tube. When the liquid level was 15 cm away from the surface of the sediment (the volume of the water column was 1178 mL), injection was stopped, and the scale was marked. All columnar samples were incubated at a constant temperature according to the in situ temperature of the reservoir, and the light and dark cycles were set to 12 h each. The specific experimental device is shown in Figure S8. Afterwards, at the specified times (0, 6, 12, 18, 24, 30, 36, 42, 48, 54, 60, 72, 84, 96, 108 and 120 h), a syringe with a volume of 50 mL was used to connect the infusion hose to collect 50 mL of overlying water from 5 cm above the sediment–water surface. The water samples were collected in 50 mL polyethylene bottles and stored in a refrigerator at 4 °C. Simultaneously, the initial filtered water sample from the original sample point was immediately added to maintain the water balance. All experiments were completed within 120 h (5 days). The reason for setting this duration is that the sediment will reach a state of adsorption or release equilibrium at approximately 120 h.

Since the inflow runoff can cause changes in the physical and chemical properties of sediment, the concentration difference between the overlying water and interstitial water promotes the exchange of N and P. Therefore, the diffusion flux can be estimated by measuring the concentration gradient between the sediment interstitial water and the overlying water. The positive value represents the release process, while the negative value represents the adsorption process. Theoretically, the molecular diffusion flux (F) is calculated according to Fick's first law, and its expression is as follows:

$$F = \left[V(c_n - c_0) + \sum_{j=1}^{n} V_{j-1}\left(c_{j-1} - c_a\right) \right] / (S \times \mathrm{t}) \quad (1)$$

where F is the average exchange flux[mg·(m^2·d)$^{-1}$]; V is the volume of overlying water in the column (L); c_n, c_0 and c_{j-1} are the mass concentration of a substance (mg·L^{-1}) at the nth, 0th (initial) and $j - 1$ th sampling; c_a is the mass concentration of the substance in the added water sample (mg·L^{-1}); V_{j-1} is the $j - 1$ sampling volume (L); S is the contact area of water and sediment in columnar samples (m^2); t is the release time (d); and the calculated nutrient release rate is 5 d average exchange flux. This formula was utilized for the investigation of total phosphorus (TP), orthophosphate (PO_4^{3-}), total nitrogen (TN), ammonia nitrogen (NH_4^+-N), nitrate nitrogen (NO_3-N) and nitrite nitrogen (NO_2-N) determination in the sediment release flux of Biliuhe Reservoir.

2.4. Sample Analysis

The static release water samples and interstitial water samples were filtered using a 0.45 μm filter membrane to determine orthophosphate (PO_4^{3-}), ammonia nitrogen (NH_4^+-N), nitrate nitrogen (NO_3-N) and nitrite nitrogen (NO_2-N). The remaining unfiltered samples were used to determine total phosphorus (TP) and total nitrogen (TN) in the water. TP, PO_4^{3-}, TN, NO_3-N, NO_2-N and NH_4^+-N in the water were obtained using a continuous flow analytical system with the Auto Analyzer III (BRAN+LUEBBE, Hamburg, Germany. From Shanghai Baozhongying Instrument Co., Ltd., Shanghai, China). TP, organic phosphorus (OP), inorganic phosphorus (IP), NaOH-extractable P (NaOH-P) and HCl-extractable P (HCI-P) in the sediments of the WLFZ were extracted and analyzed by the SMT method recommended by the European Development Framework Committee [26]. TN in the sediments of the WLFZ was determined by the soil quality-determination of total nitrogen-modified Kjeldahl method (HJ 717-2014) [27].

2.5. Data Analysis

The data consist of field monitoring data and simulation experiment data from July 2022. The data were processed using Excel 2016. Correlation analysis and principal component analysis (PCA) were performed using SPSS 22.0 (IBMSPSS Statistics, Armonk, NY, USA). Pearson correlation analysis was used to analyze the relationship between the sediment P and N release rates and the other driving factors. The principal component analysis method was employed to identify the major factors influencing P and N release rates. Principal component analysis is a statistical method to convert high-dimensional data into lower dimensional space [28]. Data were visualized using OriginPro 9.1.0 (Origin-Lab Co., Northampton, UK). ArcGIS 10.2 software was utilized for drawing related graphics.

3. Results

3.1. Nutrients in the Surface Sediments

The results indicate that TN content of surface sediments in the WLFZ of Biliuhe Reservoir ranges from 1052.52 ± 49.39 to 3520.54 ± 30.31 mg/kg, with an average value of 2600.63 mg/kg, as shown in Table 1. This trend shows a gradual increase from upstream to downstream. TP content ranged from 735.59 ± 47.92 to 2490.64 ± 28.29 mg/kg, with an average of 1380.31 ± 67.59 mg/kg, which was lower than that of N. Spatially, the content of N and P in the sediments of BLH1, located in the main stream, is low. TN and TP content gradually increases downstream from upstream. However, there is distinct heterogeneity in the spatial distribution of IP, OP, NaOH-P and HCI-P contents. Among them, OP contributes to the highest proportion of TP, followed by NaOH-P and IP. Therefore, as depicted in Table 1, it is evident that during the summer N content in the sediments of Biliuhe Reservoir is higher than that of P. Although the content of P is lower than that of N, the content of TP and other forms of P in the sediments is also high. Consequently, it can be inferred that the eutrophication primarily stems from substantial N and P contents in Biliuhe Reservoir in the summer.

Table 1. Distribution characteristics of N and P in sediments of the WLFZ.

Sampling Sites	TP (mg/kg)	TN (mg/kg)	IP (mg/kg)	OP (mg/kg)	HCI-P (mg/kg)	NaOH-P (mg/kg)
BLH 1	735.59 ± 47.92	1052.52 ± 49.39	561.89 ± 29.18	173.11 ± 13.29	98.77 ± 8.21	55.66 ± 6.35
BLH 2	1071.25 ± 23.39	2012.51 ± 21.43	193.40 ± 12.91	877.85 ± 42.36	128.75 ± 11.27	749.10 ± 35.21
BLH 3	1225.39 ± 31.47	1817.58 ± 29.35	241.25 ± 18.34	983.75 ± 53.14	133.82 ± 16.31	849.93 ± 42.64
BLH 4	2490.64 ± 28.29	3520.84 ± 30.31	585.82 ± 20.18	1904.18 ± 72.16	812.45 ± 40.24	235.43 ± 26.37

3.2. N and P at the Sediment–Water Interface

The overlying water and interstitial water within the sediment were analyzed to investigate the vertical distribution characteristics of nitrogen and phosphorus (TP, PO_4^{3-}, TN, NO_3-N and NH_4^+-N). Figures 4 and 5 present N and P values of BLH1 at the sediment–water interface. Figures S1–S3 display N and P values of BLH2, BLH3 and BLH4 at the sediment–water interface.

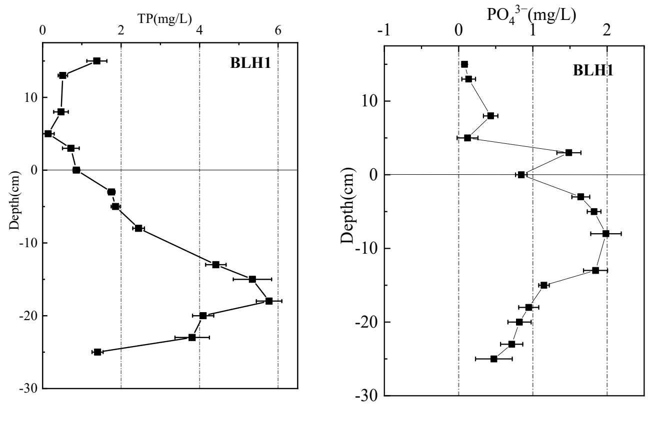


Figure 4. TP and PO_4^{3-} concentrations in overlying water and the interstitial water at the sediment−water interface (SWI) of BLH1.

It can be seen that the vertical distribution of nutrient concentrations including TP, PO_4^{3-}, TN, NO_3-N and NH_4^+-N, increases at the sediment–water interface (0 cm). As depth increases, there is a decreasing trend in nutrient concentrations with higher concentration in the interstitial water compared to the overlying water. This indicates migration and diffusion of interstitial water into the overlying water. The high concentrations of P and N in the interstitial water indicate a high release potential in the sediments. The average concentration of NO_2-N in the overlying water was higher than that in the interstitial water, indicating that NO_2-N in the sediment of the WLFZ was in the adsorption state. There are significant differences in nutrient concentration among sampling sites based on spatial distribution analysis. Further analysis of fluxes is required to determine whether sediments act as sources or sinks for nutrients.

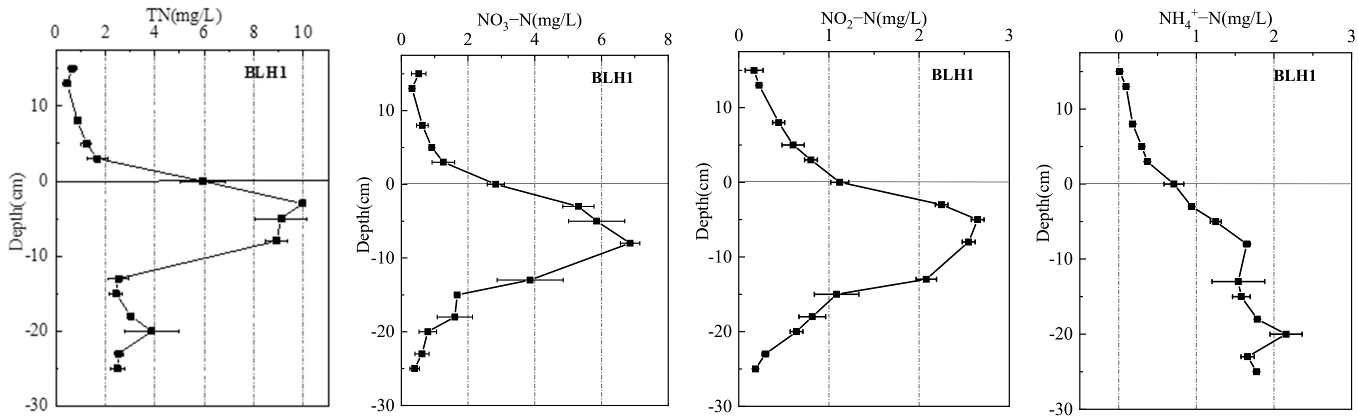


Figure 5. TN, NO_3-N, NO_2-N and NH_4^+-N concentrations in overlying water and the pore water at the sediment−water interface (SWI) of BLH1.

The vertical distribution characteristics of TP and PO_4^{3-} showed a high degree of consistency at each sampling site, with concentrations in SWI ranging from 0.03 to 10.76 mg/L and 0.06 to 9.99 mg/L, respectively. Both increased initially at the interface of 5~−5 cm and increased rapidly reached their maximum at about 0~−5 cm. The maximum TP and PO_4^{3-} of BLH3 reached 10.76 and 9.99 mg/L, respectively, and showed a decreasing trend around −10~−25 cm, rapidly decreasing to 0.23 and 0.09 mg/L. Compared to the sediment interstitial water, the average concentrations of TP and PO_4^{3-} in the overlying level were lower, measuring 1.43 ± 1.15 mg/L and 1.39 ± 1.14 mg/L, respectively. Spatially, BLH4 located in the tributary had the highest average concentration with TP and PO_4^{3-} levels at 4.54 ± 3.22 mg/L and 4.06 ± 3.19 mg/L, respectively. The P concentration of BLH1 located in the main stream is small but different from that of BLH3 and BLH4, and it experiences a rapid increase in TP concentration at −5~−12 cm followed by a gradual decrease until reaching stability.

From Figure 5, the vertical distribution characteristics of TN and NO_3-N are highly consistent, and their correlation coefficient ranges from 0.73 to 0.99, with an average value of 0.89 ($p < 0.01$), indicating a significant correlation between them. From Figure 4, it can be seen that the overlying water concentration of TN and NO_3-N is lower than that of the sediment interstitial water. TN concentrations of BLH1, BLH2, BLH3 and BLH4 were 3.71 ± 3.21, 2.92 ± 1.93, 14.09 ± 5.34 and 6.78 ± 3.83 mg/L, respectively. In terms of vertical variation, the concentrations of TN and NO_3-N in BLH1, BLH2 and BLH4 increased by about 0~5 cm. The concentrations of TN and NO_3-N in BLH3 increased in the range of −10~−15 cm. The maximum concentrations of TN and NO_3-N reached 24.10 mg/L and 19.56 mg/L, respectively. The interstitial water is significantly higher than the overlying water, suggesting a high release potential in the sediments of TN and NO_3-N. The concentrations of TN and NO_3-N in the sediment were the main factors affecting the water quality of Biliuhe Reservoir.

From Figure 5, the vertical distribution characteristics of NO_2-N increased rapidly at the interface (0~5 cm), then decreased slowly at 0~−12 cm, and finally stabilized. Compared with the sediment interstitial water, the concentrations of BLH1, BLH2 and BLH4 in overlying water were lower than that in sediment interstitial water, which were 0.56 ± 0.47, 0.77 ± 0.64 and 0.87 ± 0.54 mg/L, respectively. BLH1, BLH2 and BLH4 showed that the sediment released NO_2-N to the overlying water. The concentrations of the overlying water and the sediment interstitial water in BLH3 were 3.51 ± 1.43 and 1.95 ± 1.90 mg/L, respectively, indicating that BLH3 was opposite to the other sampling sites. The average concentration of the overlying water was significantly higher than that in sediment interstitial water, indicating that NO_2-N was mainly in the adsorption state.

From Figure 5, NH_4^+-N concentration exhibited a consistent upward trend throughout the vertical profile. BLH2 displayed a mutation point at 0~5 cm, followed by a rapid increase and subsequent decline, and then continuing to grow slowly. The mass concentration of NH_4^+-N in the overlying water was low, and the average concentration of each sampling site was 1.53 ± 0.96 mg/L. The interstitial water was significantly higher than the overlying water, which indicates a high release potential in the sediments of NH_4^+-N in the overlying water, and the concentration of NH_4^+-N in the sediment is the main factor affecting the water quality of Biliuhe Reservoir.

3.3. The Variability of Environmental Parameters and Nutrient Concentrations in the Overlying Water

The variability of environmental parameters in the overlying water is given in Figure 6. It can be observed that the WT of the water column fluctuates initially, possibly due to the difference between day and night temperatures. After 60 h, the WT gradually decreases and eventually stabilizes. The average temperatures of BLH1, BLH2, BLH3 and BLH4 are 25.41 ± 1.10, 25.44 ± 1.15, 25.52 ± 1.22 and 25.47 ± 1.19 °C. There is no significant difference in WT change between sampling sites. The change in DO can be divided into two stages: Initially, DO changed smoothly between 7.09 and 8.80 mg/L and then showed a steady downward trend at around 60 h, indicating gradual consumption of DO in the water column. During the first stage (before 60 h), average DO concentrations for BLH1, BLH2, BLH3 and BLH4 were 8.31 ± 0.86, 8.30 ± 0.38, 8.10 ± 0.64 and 7.36 ± 0.14 mg/L, respectively. In the second stage (after 60 h, including 60 h), the average DO concentrations were 4.40 ± 0.37, 6.62 ± 0.30, 6.54 ± 0.72 and 5.09 ± 1.02 mg/L, respectively. The change trend in chlorophyll-a and DO was consistent. Chlorophyll-a is an important index to reflect the number of algae. The life activity of algae is related to DO. When algae reach a certain magnitude, their abundance and activity control the change in DO in water [29]. As shown in Figure 6, chlorophyll initially remained stable over time, decreased at 60 h, and then steadily declined. The average chlorophyll-a concentrations of BLH1, BLH2, BLH3 and BLH4 were 20.31 ± 6.41, 23.01 ± 2.73, 22.54 ± 3.04 and 18.40 ± 4.59 μg/L, respectively. Turbidity gradually decreased over time due to the settling of particulate matter in the water column into the sediment. Among them, the average concentration of turbidity in each sampling site of water column was BLH4 > BLH3 > BLH2 > BLH1 (82.29 mg/L > 78.96 mg/L > 28.18 mg/L > 24.85 mg/L). Ec showed a fluctuating increase from 0 to 40 h, followed by a steady increase from 40 to 120 h. The average Ec for each sampling site of the water column is ranked as BLH3 > BLH1 > BLH2 > BLH4 (419.50 μs/cm > 419.38 μs/cm > 413.31 μs/cm > 408.63 μs/cm). The pH average values of all sampling points were ranked as BLH3 > BLH1 > BLH4 > BLH2 (7.78 > 7.77 > 7.74 > 7.73). The pH values of the water column fluctuated irregularly before 60 h. After 60 h, it decreased rapidly and then showed a steady downward trend. The pH values fluctuated from 7.52 to 7.96, with a small variation range.

The concentrations of TP, PO_4^{3-}, TN, NO_3-N and NO_2-N in the overlying water exhibited a rapid release pattern during the initial stage, reaching their maximum release at 84 h as shown in Figure 7 and Figures S4–S6. Subsequently, the flux of release gradually decreased and eventually stabilized. The release of PO_4^{3-} from BLH1 showed a rapid increase within the first 24 h, reaching its peak at 24 h, followed by a steady decrease. Sediment nutrients were primarily released into the overlying water. However, as time increased, the release rate of NH_4^+-N decreased and showed a negative flux, and the whole water column was dominated by sediment adsorption of the overlying water nutrient. Among them, the concentrations of TP and PO_4^{3-} in the overlying water of columnar sediments at each sampling site exhibited a temporal increase, while TN, NO_3-N and NO_2-N displayed fluctuating upward trends over time. The NO_2-N concentration in BLH3 and BLH4 gradually decreased over time, indicating nutrient diffusion from the overlying water to the sediment primarily through adsorption (Figures S5 and S6). NH_4^+-N concentration exhibited a fluctuating downward trend over time and was predominantly adsorbed at the sediment–overlying water interface throughout the sediment release process.

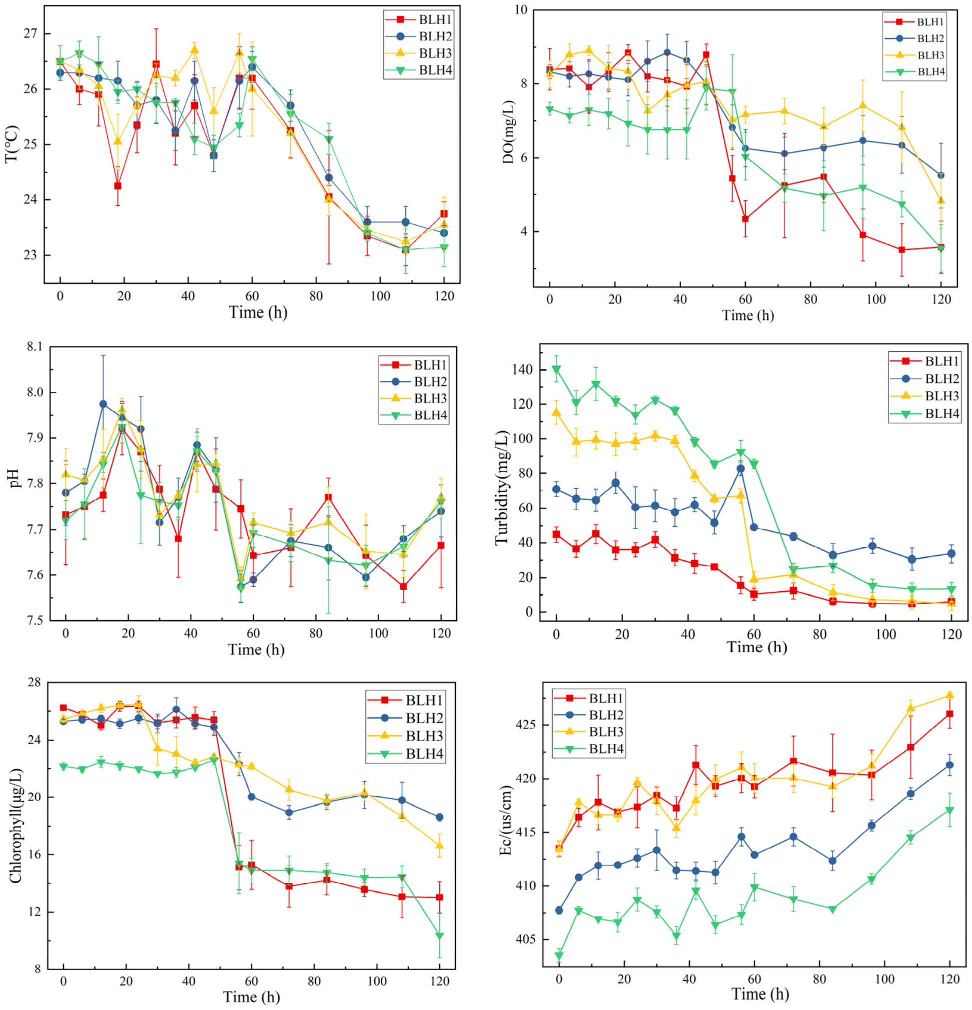


Figure 6. The variation pattern of water environment parameters in the overlying water.

The simulation experiment was conducted during summer, ensuring consistent environmental conditions with the reservoir. The indicators obtained from the experiment align with field investigation and monitoring results during summer. Therefore, it can be inferred that the simulation experiment outcomes accurately reflect N and P release after sediment overlying water in the reservoir's WLFZ under summer environmental conditions.

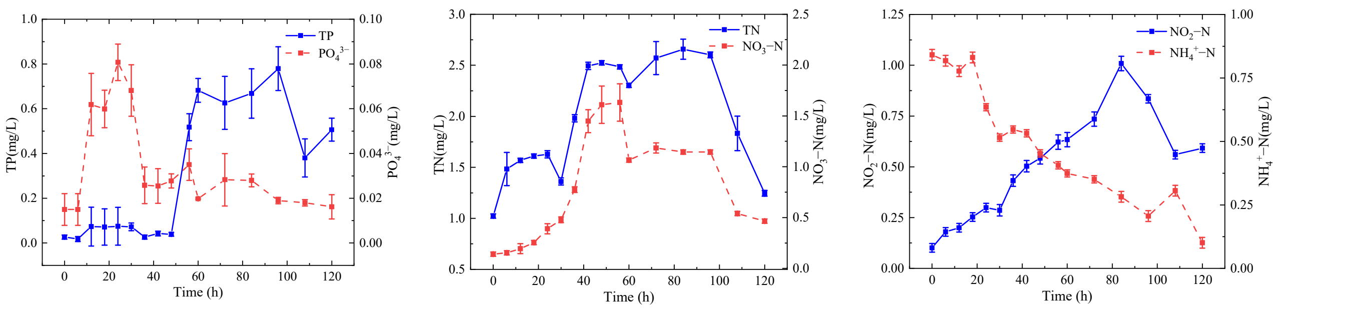


Figure 7. The temporal variation characteristics of N and P in the overlying water during the sediment incubation process of BLH1.

3.4. Deposition Release Regularity of N and P in the WLFZ

TP, PO_4^{3-}, TN, NO_3-N and NO_2-N showed positive fluxes at each site in the WLFZ, indicating nutrient release from sediments to the overlying water. Conversely, NH_4^+-N exhibited negative fluxes at each site, suggesting nutrients from the overlying water migrated towards the sediment.

The total release rates of TP and PO_4^{3-} during the static incubation experiment ranged from 0.05 to 10.21 mg·(m^2·d)$^{-1}$ and 0.03 to 6.92 mg·(m^2·d)$^{-1}$, as shown in Figure 8. Spatially, P transport regularity varied among different sampling sites, with a gradual increase in release rate from upstream to downstream. Notably, BLH4 located in a tributary of the Bajia River exhibited the largest concentration change and release rate. The release rate of TP was 7.58 ± 2.03 mg·(m^2·d)$^{-1}$, and PO_4^{3-} was 3.38 ± 2.49 mg·(m^2·d)$^{-1}$. Combined with the vertical distribution characteristics, the concentrations of N and P in BLH4 interstitial water are higher and are located downstream of the reservoir entrance. BLH4 was sampled during a field investigation with low water transparency and black, smelly sediment. It may be that domestic and agricultural sewage entered the river, resulting in a high release potential at the sampling site. Combined with the vertical distribution of nutrients in the water column, TP had a significant concentration difference (release potential) between the interstitial water and the overlying water. However, regarding its spatial distribution characteristics, the mass concentration of TP in the interstitial water (BLH4 > BLH3 > BLH1 > BLH2) was not completely consistent with the P release of sediment (BLH4 > BLH3 > BLH2 > BLH1). This indicates that the flux of P release is not completely determined by the concentration difference (release potential) between the sediment and the overlying water. Perhaps under aerobic conditions, the millimeter-level aerobic layer on the sediment surface and a diffusion boundary layer at the sediment–water surface will prevent the migration of P from the interstitial water to the overlying water. When DO decreases in the overlying water, these layers become thinner or disappear, allowing for easier diffusion of dissolved phosphorus from the interstitial water to the overlying water due to the increased concentration gradient.

As shown in Figure 9, the release rates of TN, NO_3-N and NO_2-N were 10.95~24.19 mg·(m^2·d)$^{-1}$, 0.06~13.47 mg·(m^2·d)$^{-1}$ and −2.18~10.89 mg·(m^2·d)$^{-1}$, respectively, indicating positive fluxes where nutrients of sediments were released to the overlying water. NH_4^+-N release rate ranged from −3.76 mg·(m^2·d)$^{-1}$ to 5.48 mg·(m^2·d)$^{-1}$, with an average value showing a negative flux at each site, suggesting transportation of nutrients from the overlying water into the sediment. The tributaries (BLH2, BLH3 and BLH4) were significantly higher than the main stream (BLH1), which was consistent with the trend of NH_4^+-N content in surface sediments. Notably, BLH4 displayed the highest flux. Similar to TP, NH_4^+-N had a strong concentration difference (release potential) between the interstitial water and the overlying water. The spatial distribution characteristics of NH_4^+-N mass concentration in the interstitial water (BLH3 > BLH4 > BLH2 > BLH1) and NH_4^+-N release in SWI (BLH3 > BLH4 > BLH1 > BLH2) were also different. This is because NH_4^+-N in the interstitial water can not only enter the overlying water through molecular diffusion but can also reduce its concentration in the facultative anaerobic layer of the sediment surface through the process of nitrification or ammoxidation, thus reducing the release potential of NH_4^+-N in the interstitial water.

In conclusion, the sediments in the WLFZ of Biliuhe Reservoir primarily act as a 'source' by releasing N and P into the overlying water. Considering this geographical setting, it is evident that endogenous release plays a crucial role in eutrophication within Biliuhe Reservoir. Moreover, the release flux of N and P from each tributary exceeds that of the main stream significantly. BLH4, situated at the entrance of Bajia River, exhibits substantial potential for nutrient release. Moreover, it has been observed during sampling that the water is turbid in the summer. BLH2 is located in the Geli River, where the water depth is deep and nutrients easily accumulate due to summer rainfall runoff and strong release of endogenous N and P. Additionally, BLH1 in the reservoir also has a large nutrient release potential. These areas should be focused on for controlling eutrophication in the reservoir water.

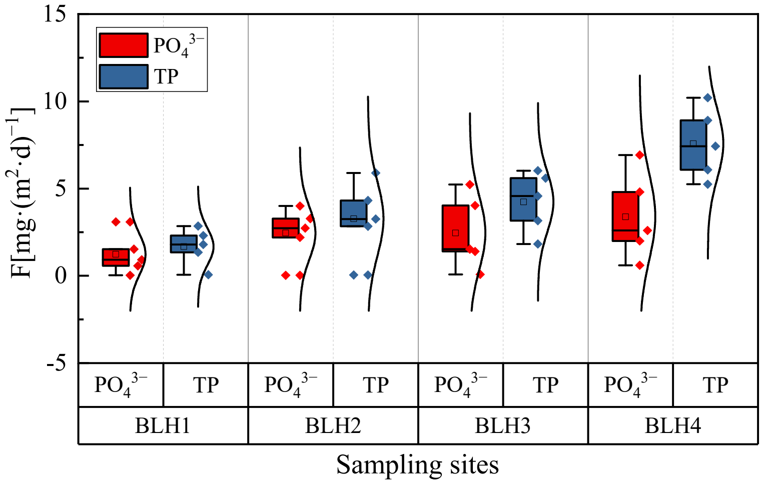


Figure 8. Releasing rates of TP and PO_4^{3-} in sediments during static incubation.

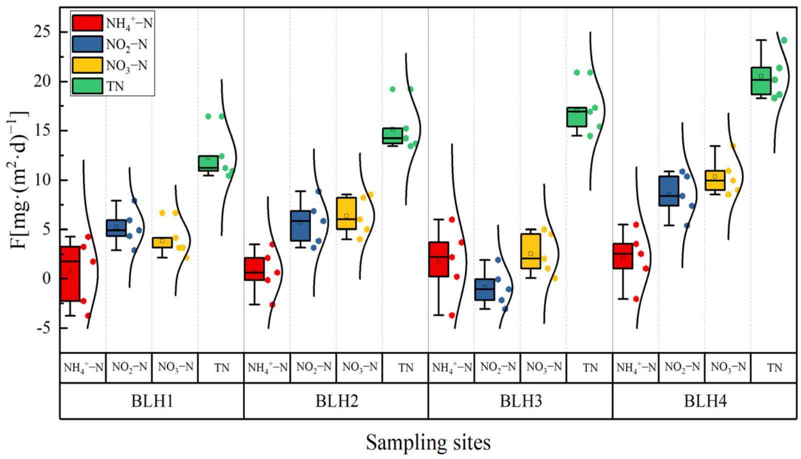


Figure 9. Releasing rates of TN, NO_3-N, NO_2-N and NH_4^+-N in sediments during static incubation.

4. Discussion

4.1. The Migration Behavior and Mechanisms of N and P in Sediments

The concentrations of N and P nutrients (TP, PO_4^{3-}, TN, NO_3-N, NO_2-N and NH_4^+-N) in the overlying water of the WLFZ of the reservoir have a high potential for diffusion with the interstitial water. Kojima et al. [30] believe that certain nutrients and trace elements present in the interstitial water of the sediment can facilitate the release of nutrients into the overlying water through the interstitial water. This is consistent with the view of our study, which also indicates that the concentration difference of nutrients in overlying water was similar to that in sediments (Figures 4 and 5). This reflects how nutrient diffusion within sediments affects the overall water quality of the reservoir area. The concentrations of TN and NO_3-N in water were higher than those in other source water reservoirs or lakes, such as Luoma Lake [31], Hengshan Reservoir [32], Fuxian Lake [33], Nansi Lake [34], Daheiting Reservoir [35] and Deer Lake [36]. The concentrations of TP and PO_4^{3-} were lower than those of N. Although the concentration of nitrogen is higher than that of phosphorus, the concentration of phosphorus cannot be ignored. Consequently, it can be inferred that the eutrophication of the Biliuhe Reservoir basin during summer primarily stems from the optimal concentration and ratio of nitrogen and phosphorus.

Microorganisms mediate the transformation of TN, NH_4^+-N, NO_3-N and NO_2-N in inorganic nitrogen components, which undergo nitrification, denitrification and ammonification under specific physical and chemical conditions. These processes are complex and changeable. Previous studies have demonstrated that molecular diffusion alone is not the dominant process controlling nutrient fluxes [37]. Cornwell et al. [38] think that microorganisms, nitrification, denitrification and ammonification conditions can also promote the diffusion of N from the sediment to the overlying water [39]. Compared with P, the effect of microbial action on its migration is more obvious. And Cheng et al. [40] think that ammonium was released from the sediment into the overlying tidal water to microbial action, with an annual mean flux of 2.74 mmol m^{-2} day^{-1}. Compared with ammonium, nitrate was mainly influxed from the overlying water into the sediment, with an annual mean flux of −2.06 mmol m^{-2} day^{-1} with microbial action. Biliuhe Reservoir is situated in a zone of flooding and drying alternation. Since mid-April, the strong light in the WLFZ caused a gradual increase in water temperature at the bottom of the stagnant layer, leading to enhanced microbial activity and a gradual decrease in DO levels. This phenomenon marks the initial stage of the summer algae outbreak [41]. The NH_4^+-N profile from sediment interstitial water (Figure 5) shows an intuitive concentration gradient between overlying water and interstitial water. In the whole 25 cm sediment profile, NH_4^+-N increased with the increase in sediment depth [42]. This aspect indicates that interstitial water can migrate and diffuse along the concentration gradient during nutrient migration in the sediment overlying water. Daniel et al. [43] also think that interstitial water can migrate and diffuse along the concentration gradient, and sediments were a net sink of −16.28 mmol N yr^{-1} for oxidized inorganic nitrogen (NO_3^- + NO_2^-). When the sediment of the WLFZ is covered with water, high concentrations of N will migrate through the interstitial water to the reservoir's overlying water, leading to a decline in reservoir water quality. On the other hand, the increase in NH_4^+-N concentration in the sediment with depth is due to the deepening of anaerobic conditions, which inhibits aerobic bacteria and anaerobic conditions. This weakens nitrification in the deep sediment while enhancing denitrification and ammoniation processes. The anaerobic environment facilitates the accumulation of NH_4^+-N, resulting in a gradual increase [44]. And Nedwell and Trimmer [45] believe that denitrification in estuarine sediments often removes a significant proportion of the dissolved inorganic nitrogen (DIN) load fluxing through an estuary and thereby plays an important role in ameliorating coastal eutrophication. In terms of spatial distribution, the migration flux of NH_4^+-N exhibited a unique distribution pattern. During the deposition release process in the WLFZ, BLH3 released the highest amount of NH_4^+-N followed by BLH4, BLH1 and then BLH2. Despite being located downstream, BLH4 did not exhibit the highest migration flux among all sampling sites. This observation contradicts with that of TN and TP migration fluxes. The reason was that BLH3 was located in the WLFZ of the agricultural planting area, where a significant quantity of loose and malodorous floating mud was discovered in the collected sedimentary column. The characteristics of loose and porous surface and high pollution promoted the conversion of organic matter to NH_4^+-N.

Compared to N, the migration and transformation of P in the flooding and drying alternation zone of the reservoir are more influenced by both the physical and chemical characteristics of the sediment and the overlying water. According to Yang et al. [46], the troph and development of the lake are influenced by the concentration of P in lake sediments. This aligns with our study's perspective. The WLFZ soil is capable of supporting a plethora of plant growth during the drying phase, with PO_4^{3-} serving as a vital nutrient source for their development and proliferation [47]. However, exposure of sediment reduces its phosphorus adsorption capacity due to drying and oxidation conditions, thereby providing phosphate for plant growth. Zhang et al. [48] indicated that the decomposition of aquatic plant debris is a key factor in the release of P from sediment even when external P is excluded. It is therefore necessary to remove plant debris from freshwater ecosystems to control the release of P from plant debris and sediment. Conversely, submerging plants during the water-covering period leads to decomposition and release of higher levels of P into the water. This suggests that plant growth in the WLFZ may indirectly promote P release from sediment into the water. Additionally, Larsen et al. [49] believe that submerged plants will consume oxygen in the sediment–water interface. The decrease in oxygen across the sediment–water interface after inundation could be another significant factor for the increase in P fluxes. The average PO_4^{3-} accounted for 82.67% of TP in the overlying water of the WLFZ of the Biliuhe Reservoir, which significantly differed from the relatively balanced TP distribution in the reservoir as a whole. Therefore, analyzing PO_4^{3-} flux of the sediments in the WLFZ is crucial to understanding this issue. A P diffusion boundary layer exists on the sediment surface, and its structure is closely related to the concentration of DO in the surface layer. Combined with the temporal changes in DO in the water column environment (Figure 6), it is evident that high DO concentration, along with an intact aerobic layer, effectively prevents the release of high concentrations of PO_4^{3-} from sediment into the water. However, under anaerobic conditions, this barrier is disrupted and a significant amount of PO_4^{3-} is released into the water column, leading to an increase in its concentration [50,51]. This is the reason why the concentration of PO_4^{3-} is higher at the sediment–water interface in the vertical direction of the water column (Figure 4). With the increase in temperature at the end of spring, the mineralization of organic matter in sediments was strengthened, so that more PO_4^{3-} was generated and released into interstitial water [52]. This also explains that PO_4^{3-} in the interstitial water profile reaches a peak only around −2 cm, and then the change tends to be stable (Figure 4). The TP concentration of the overlying water in the WLFZ of the reservoir was not significantly different, while the interstitial water of the sediment showed obvious spatial differences. The interstitial water of BLH4 has higher P concentration and is located at the entrance of Bajia River, which is a tributary of the reservoir. Field investigation of BLH4 sampling found that the water transparency was low, and the sediment appeared black and odorous. In the column release experiment, even the surface 2~3 cm of sediment completely floated in the overlying water, indicating poor sediment quality. The analysis suggests that pollutants may have entered the river, leading to increased release potential at the sampling site.

4.2. Influencing Factors of Nitrogen and Phosphorus Migrating in Deposition Release Process

The static release experiment revealed a deeper mechanism of N and P nutrient transformation, and the migration process of N and P in the flooding and drying alternation area of the WLFZ was also affected by the physical and chemical properties of the overlying water, sediment and sediment–water interface, such as WT, DO, pH, turbidity, chlorophyll-a and EC [53]. Previous research has demonstrated that DO, turbidity and chlorophyll are significant factors influencing the deposition and release of N and P [40,46,49,50]. Rippey et al. [54] think that through changes in physicochemical indicators, like water fluctuation, pH and DO levels, the formerly stockpiled P in the sediment can be released again as an endogenous source from the sediment, so as to further promote P to enter the overlying water body through the sediment–water interface. Our study also found a similar trend (Section 3.3). Therefore, based on the above discussion on the deposition release mechanism of N and P, the influencing factors of the change in N and P migration rate were analyzed.

Pearson correlation analysis and principal component analysis were conducted to examine the relationship between the release rate of N and P and water environmental factors (Table 2). The positive value indicates a positive correlation between the two variables, while the negative value suggests a negative correlation. A correlation coefficient exceeding 0.7 indicates a strong association. A correlation coefficient ranging from 0.4 to 0.7 suggests a close relationship, while a correlation coefficient ranging from 0.2 to 0.4 indicates a weak connection between two variables. The findings indicated that P of the sediment release rate in the WLFZ was significantly, negatively correlated with DO, turbidity and chlorophyll-a ($p < 0.05$) (r = −0.777, 0.870 and −0.791, respectively). The TN of the sediment release rate in the WLFZ was significantly, negatively correlated with turbidity, chlorophyll-a and DO ($p < 0.05$) (r = −0.883, −0.863 and −0.838, respectively). Furthermore, principal component analysis (Figure 10) revealed a strong correlation between PC1 and DO, turbidity and chlorophyll-a, as evidenced by their high load coefficients on PC1. PC2 was strongly associated with WT and pH, as evidenced by the large loading coefficients of these variables on PC2. Combined with Table S2, the component score coefficient matrix was utilized to identify the driving factors in each principal component. During this analysis, DO, turbidity and chlorophyll-a were found to contribute significantly to the first principal component with eigenvectors of 0.972, 0.965 and 0.942, respectively. In general, DO, turbidity and chlorophyll-a were more important factors affecting the release of N and P in the sediments of the WLFZ. This result can be explained from the following aspects:

Table 2. Pearson correlation coefficients between TN/TP and water environmental factors.

	WT	DO	pH	Turbidity	Chlorophyll-a	Ec
TN	−0.537	−0.838 **	−0.715	−0.883 **	−0.863 **	0.555 *
TP	−0.693	−0.777 **	−0.704	−0.870 **	−0.791 **	0.605 *

Notes: * Correlation is significant at the 0.05 level (two-tailed). ** Correlation is significant at the 0.01 level (two-tailed).

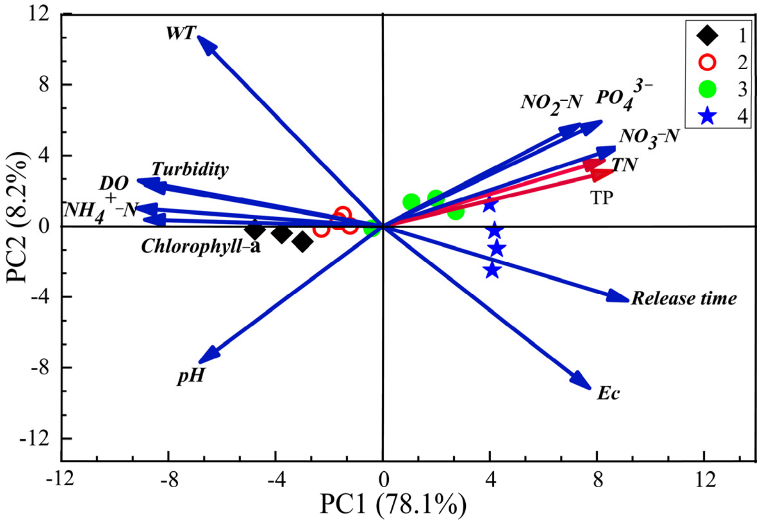


Figure 10. Principal component analysis (PCA) (all impact factors in the PC1−axis and PC2−axis ordination planes. The arrows represent the feature vector of the influence factors. The rhombus, hollow circle, solid circle and pentagram represent the grouping and sorting according to principal component analysis).

Firstly, DO concentration in the overlying water was closely related to N and P release fluxes of the sediments in the WLFZ (p < 0.001) (Figure S7a,d). When the drying sediment in the WLFZ becomes submerged, the organic matter and microorganisms are released into the overlying water. The high organic matter becomes submerged, resulting in a gradual decrease in DO concentration in the overlying water within the WLFZ [55,56]. Then, the reduction in DO further facilitates the dissolution of N and P, leading to an increase in their release from sediments [57]. Jin et al. [58] also think that phosphorus can be adsorbed from overlying water to sediments with high nutrient loading under low dissolved oxygen conditions. Therefore, when the exposed sediments within the WLFZ are inundated with water, there is an elevation in N and P loading within the sediment. This increase is particularly significant in northern China during July. Furthermore, the DO concentration in water indirectly regulates microbial activity, affecting nitrification and denitrification processes. Hardison et al. [59] propose that DO is a pivotal factor influencing both nitrification and denitrification processes. Consequently, DO plays a crucial role in modulating sediment nitrogen release. Under aerobic conditions, nitrifying bacteria converts ammonia to nitrate. When the DO concentration of the overlying water is low, denitrification becomes dominant. Hou et al. [60] demonstrated significant alterations in the nitrogen cycle, particularly nitrification, denitrification and mineralization: furthermore, the presence of oxygen also affects nitrogen diffusion. When the WLFZ is dry, the nitrate removal rate is more than 90%. At this time, nitrite is reduced to nitrogen (N_2) and removed from the sediment–water system. Many scholars have discovered that DO plays a crucial role in controlling the diffusion and adsorption of NH_4^+-N [61]. As depicted in Section 3.3, NH_4^+-N release from sediments is low when the DO concentration is high but increases under low DO conditions [62]. This observation provides evidence for the negative correlation between the DO concentration of the overlying water and the N release from sediments (Figure S7d). At the 120th hour, the average DO concentration in the overlying water at all four sampling sites reached its lowest value (4.67 mg/L), while the diffusion flux of NH_4^+-N peaked at 5.48 mg·(m^2·d)$^{-1}$. When the average value of DO in the overlying water reached its highest point (8.38 mg/L) at 18 h, the diffusion flux of NH_4^+-N significantly decreased. Therefore, by maintaining a certain DO concentration in the overlying water of the WLFZ, effectively inhibits ammonium diffusion [63].

Turbidity is characterized by the suspended matter concentration and particle size in the water. Although TN concentration gradually decreases over time (Section 3.3), turbidity does not affect the release of TN from sediment (Figure S7e). Unlike N, P in water is easily adsorbed by sediment particles. The turbidity and suspended solids concentration of water significantly influence the distribution of P forms in water (Figure S7b). In the WLFZ of the reservoir, wave disturbance enhances suspended solids transport in the overlying water, resulting in higher concentrations of TP and total particulate phosphorus (TPP) as suspended solids increase. Gao et al. [64] showed that heavy rainfall increased the turbidity of the water and had a significant effect on P-forms at the entrance of Taihu Lake. Bengtsson et al. [65] proposed that hydrodynamic disturbance is a recognized factor influencing the release of endogenous P, facilitating sediment resuspension and promoting P release. Under conditions of hydrodynamic disturbance, there may be an increase in both the overlying water and the amount of P released from sediments. These views are consistent with the results of our study. The response of TPP concentration was the fastest, and the peak appeared on the day of heavy rainfall. It is evident that the turbidity and suspended matter concentration of water in each basin were increased, with a pronounced adsorption effect on P. This view can prove that the content of turbidity in the overlying water will directly affect the migration and release of P in sediments in the WLFZ.

The chlorophyll-a concentration in the overlying water is an important indicator of the release of N and P from the sediment in the WLFZ of Biliuhe Reservoir. The decline of algae in the overlying water with incubation time leads to a change in DO concentration, indirectly promoting P release from sediment. Our research also highlights that DO concentration is a key factor affecting P release from sediments. It controls the adsorption and release of various forms of P in sediments by controlling the oxidation-reduction potential (ORP) of water, the species and activity of aquatic organisms and the mineralization process of organic matter [66]. On the other hand, the decline in cyanobacteria exacerbates the release of bio-available P in sediments, which has a significant impact on endogenous P release in overlying water. The decline in algae makes the sediments in an extremely anoxic environment, resulting in the decrease in iron-bound phosphorus (Fe-P), calcium-bound phosphorus (Ca-P) and aluminum-bound phosphorus (Al-P) contents in the sediments. This shows that the death deposition of algae has a great influence on the release of bio-available P [67]. Qian et al. [68] also believe that microbial populations and water quantities can influence the dissolution, transformation, migration, aggregation and deposition of P. By fitting the chlorophyll content of the overlying water with the release of N and P from sediments, a significant relationship between these two variables was established ($p < 0.001$) (Figure S7c,f). At its peak value of 25.13 μg/L at 18 h, the chlorophyll concentration in the overlying water corresponded to relatively low TN and TP fluxes of only 1.23 and 0.15 mg·(m^2·d)$^{-1}$.

Therefore, in the process of ecological maintenance and construction in the WLFZ of the reservoir, maintaining a certain DO concentration in the overlying water effectively inhibits NH_4^+-N diffusion by controlling nitrification and denitrification. Simultaneously, turbidity and chlorophyll-a are the main environmental factors affecting the transformation and migration of N and P in the sediments of the WLFZ. The control of turbidity and chlorophyll-a in the water should be strengthened. In addition, the release of sediments in the WLFZ is also affected by soil type, pH, ORP and organic matter concentration. At the same time, the import of exogenous P in the sediments of the estuary should be avoided, and effective measures should be taken to control exogenous pollution and dredging in the WLFZ. Effective control measures are still crucial for preventing further long-term adsorption in the water from the sediments in the WLFZ of Biliuhe Reservoir, as well as controlling the accumulation of endogenous N and P and input of exogenous pollutants.

4.3. Countermeasures and Suggestions for Sediment Eutrophication in the WLFZ of the Reservoir

Comparing NH_4^+-N and PO_4^{3-} migration rates of sediment in the WLFZ of Biliuhe Reservoir with those in previously published studies (Figure 11, Table S3), it is evident that the internal load in the WLFZ of Biliuhe Reservoir is relatively high. Specifically, PO_4^{3-} has a higher migration rate than the typical lakes and reservoirs studied before. Although the release of NH_4^+-N in the WLFZ of Biliuhe Reservoir is higher than other reservoirs, it falls within a similar range. The water quality of Biliuhe Reservoir was compared with WHO drinking water quality standards [69] and standards for drinking water quality in China [70]. As indicated in Table 3, among these crucial water quality indicators, the water quality of Biliuhe Reservoir is essentially compliant with both WHO standards and China's drinking water quality standards. The coliform bacteria slightly exceeded the standard, but it remained within the manageable range and did not affect the drinking water quality of Biliuhe Reservoir. With summer approaching, the increased bottom water temperature and lower bottom DO in the WLFZ also means a greater release risk. Therefore, it can be concluded from the results that the SWI nutrient release flux in the WLFZ of the reservoir has a greater release risk than the SWI in the reservoir. In summer, the inflow runoff easily carries nutrients, resulting in an increase in internal load.

For this reason, the Management Bureau Biliuhe Reservoir in Dalian City established a multi-stage impermeable submerged dam perpendicular to the flow direction at the appropriate location of the inflow of the WLFZ in 2019. It is utilized to intercept surface runoff and underground seepage, elevate the water level of the main channel, mitigate flow velocity in the main channel, enhance hydraulic retention time and purification capacity of the inflow of the WLFZ for suspended solids and pollutants carried by inflow runoff, prevent continuous erosion of the main channel by runoff, and provide more favorable flow conditions for aquatic plant growth. The findings of our study, however, indicate that the release of N and P from the sediment in the reservoir within the WLFZ cannot be disregarded. Therefore, for the pollution of the WLFZ of the reservoir, common restoration methods such as algae salvage, ecological restoration and sediment dredging can be used to control the eutrophication of the reservoir. For areas with high internal release fluxes, such as BLH3 and BLH4 of the tributary (Figures 8 and 9), dredging can be used to remove internal contaminants. Ecological restoration can be implemented in areas with low fluxes, such as BLH1 situated in the main stream. However, considering the persistent impact of residential areas on water quality in the three tributaries of the reservoir, it is crucial to enhance external pollution control measures and implement integrated management strategies for both internal and external sources in order to effectively restore the water quality of this eutrophic reservoir.

Table 3. The water quality of Biliuhe Reservoir was compared with WHO drinking water quality standards and standards for drinking water quality in China.

Indicators	WHO [69]	Standards for Drinking Water Quality in China [70]	Biliuhe Reservoir
pH	6.5–8.5	6.5–8.5	8.3
NO_3^- (measured by N)	10 mg/L	--	2.14 mg/L
NH_4^+ (measured by N)	1.5 mg/L	0.5 mg/L	0.08 mg/L

Table 3. *Cont.*

Indicators	WHO [69]	Standards for Drinking Water Quality in China [70]	Biliuhe Reservoir
Cu	1.0 mg/L	1.0 mg/L	0.01 mg/L
Zn	5.0 mg/L	1.0 mg/L	0.02 mg/L
F	1.5 mg/L	1 mg/L	0.03 mg/L
Cd	0.005 mg/L	0.005 mg/L	0.0005 mg/L
Cr	0.05 mg/L	0.05 mg/L	0.004 mg/L
Pb	0.05 mg/L	0.01 mg/L	0.0025 mg/L
$(CN)_2$	0.1 mg/L	0.05 mg/L	0.004 mg/L
SO_4^{2-}	400 mg/L	250 mg/L	26.38 mg/L
Fe	0.3 mg/L	0.3 mg/L	0.003 mg/L
Mn	0.1 mg/L	0.1 mg/L	0.001 mg/L
Turbidity	5 NTU	1 NTU	1.13 NTU
Coliform bacteria	It cannot be detected in any 100 mL water sample.	It cannot be detected in any 100 mL water sample.	23 MPN/100 mL
Total hardness (measured by $CaCO_3$)	500 mg/L	450 mg/L	89.6 mg/L
Chromaticity	15 TCU	15 TCU	5 TCU

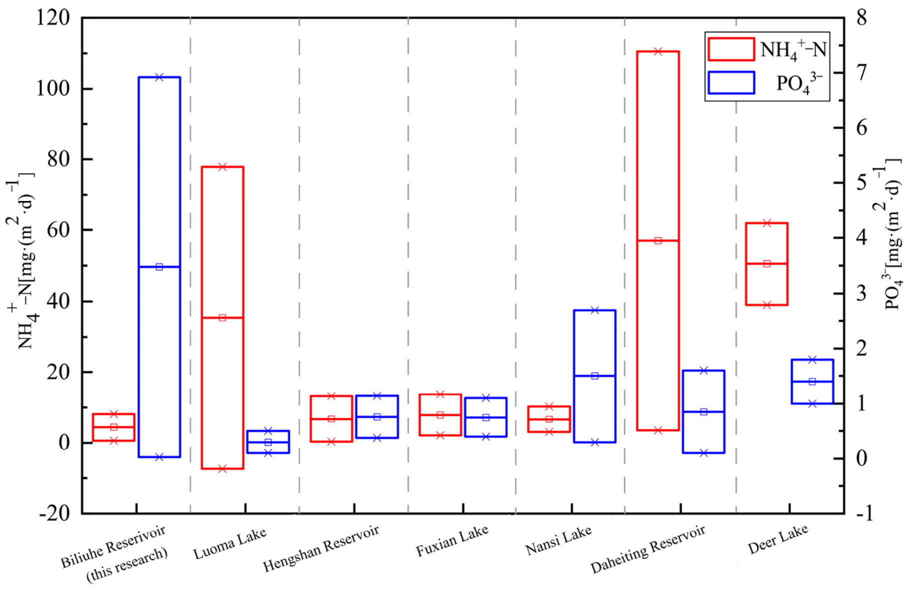


Figure 11. Comparison of fluxes in NH_4^+-N and PO_4^{3-} in previously published studies [31–36].

5. Conclusions

Through the analysis of N and P distribution characteristics and release behavior of N and P in the sediment of the WLFZ in Biliuhe Reservoir, the following can be concluded:

(1) The sediment in NH_4^+-N acts as the primary source of nutrients for the overlying water, with the concentration of NH_4^+-N in sediments playing a crucial role in determining the water quality of Biliuhe Reservoir. The average concentration of NO_2-N in the overlying water was significantly higher than that in the interstitial water, indicating predominant adsorption of NO_2-N in the sediments of the WLFZ. Conversely, TP and PO_4^{3-} demonstrate limited migration and diffusion potential, indicating that elevated concentrations of N pollution are the primary driver of eutrophication in Biliuhe Reservoir during summer.

(2) The sedimentary flux of nitrogen and phosphorus released by the main stream is lower than that of the tributary. The findings demonstrate that the water diversion project situated in the main stream has a scouring effect on the sediments of the WLFZ at this specific location, resulting in lower nutrient content within the sediments of the main stream compared to its tributaries. We hypothesize that the implementation of the water diversion project may impact the release of sediment nutrients in the WLFZ,

leading to a need for reassessment of N and P release fluxes from drinking water reservoirs worldwide in order to fully comprehend endogenous pollutant releases.

(3) DO, turbidity and chlorophyll-a are the primary factors driving N and P release in sediments within the WLFZ. The submergence of exposed sediments, particularly within the WLFZ, will lead to an increase in N and P loading within the sediment due to microbial respiration and organic matter decomposition. Maintaining a specific dissolved oxygen concentration in the overlying water of the WLFZ suppresses the diffusion of sediment nutrients.

(4) Ecological restoration can be implemented in areas with low fluxes. The high release fluxes from the three tributaries can be managed through sediment dredging and algae harvesting in affected areas. This study highlights the influence of environmental factors on the transformation and diffusion of source N and P in the WLFZ of a drinking water reservoir, providing a robust theoretical foundation for effectively managing N and P pollution in aquatic systems within this region.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/w15203659/s1, Figure S1: Nutrient concentrations in overlying water and the pore water at the sediment–water interface (SWI) of BLH2. Figure S2: Nutrient concentrations in overlying water and the pore water at the sediment–water interface (SWI) of BLH3. Figure S3: Nutrient concentrations in overlying water and the pore water at the sediment–water interface (SWI) of BLH4. Figure S4: The temporal variation characteristics of N and P in the overlying water during the sediment incubation process of BLH2. Figure S5: The temporal variation characteristics of N and P in the overlying water during the sediment incubation process of BLH3. Figure S6: The temporal variation characteristics of N and P in the overlying water during the sediment incubation process of BLH4. Figure S7: The correlation between water environmental factors and the release of N and P in sediments. Figure S8: Schematic diagram of experimental apparatus. Table S1: The porosity and grain size fractions of sediment core in Biliuhe Reservoir. Table S2: Component score coefficient matrix of factors affecting nutrient salt release rates of the sediment–water interface. Table S3: Nutrient fluxes in previously published studies across the sediment–water interface around the world. Table S4: Abbreviations and their specific meanings. Supplement of experimental scheme.

Author Contributions: Conceptualization, methodology, validation and writing—original draft, W.L.; conceptualization, funding acquisition and writing—review and editing, S.X.; funding acquisition, experiment and writing—review and editing, X.C.; experiment and drawing, D.H.; data curation, B.M. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the National Natural Science Foundation of China (Grant no. 42077348 and Grant no. 51879031).

Data Availability Statement: Data will be made available upon request.

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

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