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Forms of Nitrogen and Phosphorus in Suspended Solids: A Case Study of Lihu Lake, China

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Abstract: Suspended solids are an important part of lake ecosystems, and their nitrogen and phosphorus contents have a significant effect on water quality. However, information on nitrogen and phosphorus forms in suspended solids remains limited. Therefore, a case study was conducted in Lihu Lake (China), a lake with characteristically high amounts of suspended solids. Nitrogen and phosphorus speciation in suspended solids was analyzed through a sequential extraction method. We also evaluated the sources of various forms of nitrogen and phosphorus and their different effects on eutrophication. The total nitrogen (TN) content was 758.9–3098.1 mg/kg. Moreover, the proportions of various N forms in the suspended solids of the study areas were ranked as follows: Hydrolyzable nitrogen (HN) > residual nitrogen (RN) > exchangeable nitrogen (EN). Total phosphorus (TP) ranged from 294.8 to 1066.4 mg/kg, and 58.6% of this TP was inorganic phosphorus (IP). In turn, calcium (Ca)-bound inorganic phosphorus (Ca-Pi) was the main component of IP. The correlation between various nitrogen and phosphorus forms showed that there were different sources of suspended nitrogen and phosphorus throughout Lihu Lake. Correlation analysis of water quality indices and comparative analysis with surface sediments showed that in Lihu Lake, the dissolved nitrogen and phosphorus contents in water were influenced by sediment through diffusion, while particle phosphorus content in water was influenced by suspended solids through adsorption; however, due to the higher phosphorus contents in suspended solids, we should pay more attention to the impact of suspended solids.

Keywords: suspended solids; nitrogen; phosphorus; form analysis; Lihu Lake

1. Introduction

Eutrophication is among the most important threats to global surface water quality. Currently, more than 75% of closed water bodies worldwide present some degree of eutrophication [1–3]. According to the Organization for Economic Cooperation and Development (OECD), “eutrophication” is defined as an undesirable degradation in water quality associated with increases in the productivity of algae and aquatic plants, which is caused by increases in nutrient concentrations and affects the beneficial uses of water. As socioeconomic development continues to grow, the term “eutrophication” is commonly used to refer to events linked to anthropogenic activities, typically within a short time scale (i.e., hours, days, months, years) [4,5]. Eutrophication can cause great damage to aquatic ecosystems and has adverse effects on aquatic products, water services, human health, and economic activities in surrounding areas [6–8]. The mechanisms of eutrophic formation and diffusion have been studied since the 1970s. Nitrogen (N) and phosphorus (P) are the main growth-limiting nutrients for algae, bacteria, protozoa, and other aquatic organisms that inhabit freshwater ecosystems [9–11]. However, the excessive input of these nutrients may accelerate the eutrophication process in the affected water

bodies. Therefore, the effects of nitrogen and phosphorus on eutrophication have been studied both in water and sediments.

Although external inputs are the main factor leading to the eutrophication of lakes, the biochemical reaction of nitrogen and phosphorus at the sediment/suspended solid water interface has an impact on the nutrient contents in water. Different forms of nitrogen and phosphorus play different roles in biogeochemical cycles and, therefore, contribute differently to eutrophication [12,13]. In general, nitrogen and phosphorus are converted into different molecular forms at the sediment/suspended solid water interface. Nitrogen in sediments and suspended solids can be divided into convertible and non-convertible forms. Among them, convertible nitrogen participates actively in the nitrogen cycle [14]. On the other hand, phosphorus in sediments/suspended solids mainly exists in adsorbed, organic, iron-/aluminum-bound, and calcium-bound forms. However, only bioavailable nitrogen and phosphorus can promote algae growth and lead to eutrophication [15]. In recent years, most studies have focused on the forms of nitrogen and phosphorus that are most common in sediments. However, few studies have characterized these elements in suspended solids.

Suspended solids (SS) are among the most important elements of freshwater ecosystems, as they represent a substrate for nutrients to bind to [16,17]. The re-release of nitrogen and phosphorus in suspended solids is also one of the most important factors affecting the eutrophication of lakes. Suspended solids can easily adsorb contaminants (e.g., nutrients, heavy metals, organic contaminants) [18–21]. Moreover, suspended solids can exchange substances with water bodies during re-suspension, or reincorporate into the sediments after flocculation and sedimentation. On the other hand, the degradation of nutrients in suspended solids during suspension may lead to an accelerated migration of biogenic elements into the water bodies. These migrated elements can ultimately supplement the nutrients that are often depleted in summer due to blue-green algae blooms, and thus perpetuate algae bloom cycles. Therefore, studying the forms of nitrogen and phosphorus in suspended particulate matter is critical in establishing a theoretical basis for lake eutrophication control.

Taihu Lake, located in Jiangsu and Zhejiang province (i.e., the most economically developed areas of China), is among the five largest freshwater lakes in China. It represents the main drinking water source for Wuxi and Suzhou cities in Jiangsu province, and is one of the main water supplies for Shanghai and East Zhejiang province. However, with rapid urbanization, Taihu Lake has experienced numerous water pollution problems since the 1980s, particularly eutrophication and subsequent cyanobacterial blooms. Lihu Lake, a bay located in Wuxi City, is a typical shallow lake, with an average water depth of only 2.25 m [22,23]. Wind and wave disturbances are prevalent and affect the water dynamics of the lake. Therefore, although water quality has been greatly improved due to the implementation of aquatic environment treatment projects, the concentration of suspended solids in the lake remains high. Large amounts of SS not only reduce the transparency and affect the underwater light conditions, but are also a great challenge for the drinking water of Wuxi City. However, previous studies on Lihu Lake mainly focused on the water quality [24], nitrogen and phosphorus forms in the sediment [25,26], etc. Although Lihu Lake has been subject to a series of abatement actions to restore the ecological environment, the content of suspended solids in this lake is still high, and the eutrophication still exists. Therefore, the study of nitrogen and phosphorus forms in suspended solids is of great significance for the further ecological restoration and environmental management of Lihu Lake. However, there are few studies on the forms of nitrogen and phosphorus in suspended solids of Lihu Lake. In this study, a nitrogen and phosphorus fractionation extraction approach based on improvements to previously reported methods was used to study the forms and spatial distributions of nitrogen and phosphorus in suspended solids in Lihu Lake. Particularly, this study sought to characterize the migration and transformation of nutrients between the water–SS and SS–sediment interfaces based on the following aspects: (1) Contents and spatial distribution of nitrogen and phosphorus forms in suspended solids, (2) correlations between all of the nitrogen and phosphorus forms in overlying water, suspended solids, and surface sediments, and (3) comparison between the nitrogen and phosphorus contents in suspended solids and surface sediments.

2. Materials and Methods

2.1. Study Area

Lihu Lake (120.22° E–120.29° E, 31.48° N–31.55° N) is located north of Taihu Lake, with a 6 km length from east to west and a 0.3–1.8 km width, covering an 8.6 km² surface. The lake is connected to the Meiliang Bay of Taihu Lake, the Beijing-Hangzhou Grand Canal, and Gonghu Bay, which is relatively independent but still a part of Taihu Lake. In general, the overall flow rate of the study area is low. In the 1950s, Lihu Lake exhibited medium nutrient levels and was rich in aquatic plants and fisheries resources. Since then, the lake has provided drinking water for the entire city of Wuxi [27]. However, since the 1970s, the locals reclaimed land from the lake, and large-scale aquaculture farms were constructed. Additionally, vast amounts of domestic and industrial sewage from the surrounding areas began to be discharged into the lake. As a result, the total nitrogen (TN) and total phosphorus (TP) concentrations increased, and the water quality of the lake and its inflow rivers deteriorated rapidly, which has led to severe eutrophication. In 2001, the annual mean permanganate, total nitrogen (TN), total phosphorus (TP), and chlorophyll-a (*Chl.a*) index values were 7.80, 6.38, 0.20, and 0.08 mg/L, respectively, which were 1.39, 1.93, 2.44, and 2.61 times higher than the overall historical indices of Taihu Lake [28]. Therefore, starting in 2003, the Wuxi City government banned aquaculture, relocated the residents around the lake, and eliminated pollution sources to prevent further eutrophication of Lihu Lake. The central and local governments jointly implemented the “environmental dredging and ecological reconstruction project for heavily polluted water” in Lihu Lake, which entailed environmental dredging, vegetation reconstruction, and gate control or blockage of contaminated rivers. The water quality of Lihu Lake has since been maintained. However, water transparency and suspended solids have not improved significantly, and thus, nutrients in suspended solids may still aggravate the eutrophication process.

Nitrogen and phosphorus are the main pollutants affecting the water quality of Lihu Lake, and these contaminants originate from many external sources, as well as from endogenous release. Given that Lihu Lake is virtually isolated from the surrounding rivers by gates and dams, there is no water exchange between the lake and the surrounding rivers. The main sources of nitrogen and phosphorus in Lihu Lake are mainly internal and external sources through dry and wet deposition.

In this study, Lihu Lake was divided into four zones (A, B, C, and D), based on the physical boundaries within the study area (Lihu Dikey, Baojie Bridge, and Lihu Great Bridge; Figure 1). Zone A used to contain a large number of fish ponds, which seriously polluted the lake, after which the surface sediments were removed by draining the lake water. Zone B was known as the “western Lihu Lake” before the implementation of comprehensive remediation measures via mechanical dredging. Moreover, aquatic vegetation reconstruction was implemented offshore. Zone C, bordered by the Baojie Bridge to the west and Lihu Great Bridge to the east (including Changguangxi Wetland Park), and also implemented the comprehensive coastal improvement project. Zone D, with more residential areas offshore, exhibits the worst water quality in Lihu Lake.

Figure 1. Location and sampling sites in Lihu Lake.

2.2. Sampling and Sample Preparation

Suspended solids and surface sediment samples from 28 sites (Figure 1) in Lihu Lake were collected in October 2019. A total of 50 L of water at a 0.5 m depth were collected from each sampling site into a clean and light-proof plastic sampling bottle which had been previously rinsed with Milli-Q. Water samples were filtered with glass fiber filters (GF/F) (Whatman, Maidstone, Kent County, U.K.) (these filters were pre-soaked in 1:1000 (v:v) HCl for 24 h, then washed to neutral pH with Milli-Q water, and finally dried at 400 °C for 5 h) in situ to obtain wet suspended solid samples. These samples were then sent to the laboratory within 24 h at −4 °C and vacuum freeze-dried at −50 °C for 72 h, then stored in dark, sealed bottles at −4 °C until they were analyzed. At each site, the surface sediment samples were collected using a Petersen dredge. These samples were stored in clean black polythene bags at −4 °C before being sent to the laboratory, vacuum freeze-dried at −50 °C, and ground through a 100 mesh sieve. The prepared samples were also stored in light-proof glass sealing bottles at −4 °C. In the field, water samples were filtered through pre-weighted filters (GF/F) (Whatman, Maidstone, Kent County, U.K.) to measure the suspended solid contents.

2.3. Sample Analysis

2.3.1. Nitrogen Forms

Based on sequential extraction, the nitrogen forms in suspended solids were divided into exchangeable nitrogen (EN), acid-hydrolyzable nitrogen (HN), and residual nitrogen (RN). The sequential extraction steps for nitrogen forms and the measurement methods for each index are illustrated in Figure A1 (Appendix A).

2.3.2. Phosphorus Forms

According to the absorption and desorption characteristics of each phosphorus form in suspended solids, as well as their stability once combined with the suspended solids, and based on our improvements of previously published sequential extraction methods for phosphorus, our study divided inorganic phosphorus into weakly adsorbed inorganic phosphorus (WA-P_i), potentially active inorganic phosphorus (PA-P_i), iron (Fe)- or aluminum (Al)-bound inorganic phosphorus (Fe/Al-P_i), and calcium (Ca)-bound inorganic phosphorus (Ca-P_i). Moreover, the organic phosphorus forms were divided into weakly absorbed organic phosphorus (WA-P_o), potentially active organic phosphorus (PA-P_o), medium active organic phosphorus (MA-P_o), and non-active organic phosphorus (NA-P_o). Among these, MA-P_o includes medium active organic phosphorus, which can be extracted with a sodium hydroxide (NaOH) solution (MA_{NaOH-P_o}), as well as non-active organic phosphorus, which can be extracted with a sodium hydroxide (NaOH) solution (NA_{NaOH-P_o}), and residual phosphorus

(R-P_o). The sequential phosphorus extraction steps and the measurement methods for each index are shown in Figure A2 (Appendix A).

Nitrogen and phosphorus forms in the surface sediments were also extracted according to the aforementioned method. The measurement method of each index in the overlying water, suspended solids, and surface sediments was performed as described by Wang [25] and Wang [26].

2.4. Quality Assurance and Quality Control (QA/QC)

The analytical data quality was assessed by implementing quality assurance and quality control (QA/QC) procedures, including analysis of reagent blanks, duplicate samples, and standard reference materials (GSD-3a) for each sample batch. The analytical precision for replicate samples was within $\pm 10\%$ and the measurement errors between determined and certified values were less than 5%.

Statistical analyses of mean values and standard deviations were performed using the SPSS 22.0 software (IBM, Armonk, USA). The Kriging method was employed using ArcGIS 10.2 (ESRI, Redlands, USA) and Surfer 11 (Golden Software, Golden, USA) to map the spatial distribution of nitrogen and phosphorus forms. Other experimental data graphs were created with Excel 2010 (Microsoft, Redmond, USA) and Origin 2020 (OriginLab, Northampton, USA).

3. Results and Discussion

3.1. Spatial Distribution of Total Suspended Solids

The term “suspended solids” (SS) generally refers to plankton and inanimate particles suspended in water. Notably, the total suspended solid (TSS) content is not only positively correlated with nutrient levels, but is also positively correlated with total phosphorus (TP) contents in water. Therefore, TSS is often used as a reference index to reflect the level of nutrients in water. In 65 oligotrophic lakes in the USA and Argentina, the average annual mean TSS value was only 1.00 mg/L [29], whereas this value has been reported to be as high as 7.3 mg/L in 86 eutrophic shallow lakes in Europe [30]. However, the average TSS value in highly eutrophic shallow lakes in Denmark was as high as 22 mg/L [31]. As shown in Figure 2, in this study, the average TSS value in Lihu Lake was 27 mg/L, which is similar to that of 15 highly eutrophic shallow lakes in Denmark and West Lake in Hangzhou (25.02 mg/L) [32]. Nonetheless, the aforementioned value was lower than those of Taihu Lake (32.32 mg/L) [33] and Chaohu Lake (43.61 mg/L) [34], which are more eutrophic.

Figure 2. Spatial distribution of total suspended solids (TSS) in Lihu Lake.

Since Lihu Lake is isolated from the surrounding rivers by gate and dams, the particle solids from the surrounding rivers can be ignored. Therefore, planktonic algae proliferation, residues of

aquatic plants, and sediment resuspension are the main factors influencing the TSS concentration in water. Due to an ecological reconstruction project, a considerable area of submerged plants has been planted in Zone A, and the contents of SS in these areas are lower than those in other areas. The main reason is that the submerged plants can not only adsorb the suspended solids, but also compete with phytoplankton for nutrients and light, thus inhibiting the growth of algae further.

As summarized in Table A1 (Appendix A), the particulate nitrogen (PN) and particulate phosphorus (PP) fractions were 0.06–0.71 and 0.04–0.21 mg/L, respectively, with average values of 0.35 and 0.11 mg/L, accounting for 65.03% and 82.63% of the total nitrogen and total phosphorus contents, respectively. Moreover, the TSS value was found to have a positive correlation with the concentrations of PP ($r = 0.260$, $n = 28$) and TP ($r = 0.331$, $n = 28$) (Figure A3).

3.2. Spatial Distribution of Nitrogen Forms in Suspended Solids

3.2.1. Spatial Distribution of Exchangeable Nitrogen (EN) in Suspended Solids

Figure 3 illustrates the spatial distribution characteristics of the exchangeable nitrogen forms in suspended solids of Lihu Lake. Exchangeable nitrogen in suspended solids mainly includes exchangeable $\text{NH}_4^+\text{-N}$ (E- $\text{NH}_4^+\text{-N}$), $\text{NO}_3^-\text{-N}$ (E- $\text{NO}_3^-\text{-N}$), and exchangeable soluble organic nitrogen (SON). Exchangeable nitrogen in suspended solids can maintain dynamic equilibrium with the overlying water and surface sediments through adsorption/desorption, ion exchange, and biological disturbances [35]. Importantly, it represents a nutrient source that can be directly utilized by primary producers in lakes [36]. Therefore, the amounts of EN in SS play an extremely important role in the nitrogen cycle of suspended solids.

Figure 3. Spatial distribution of exchangeable nitrogen (EN) in suspended solids of Lihu Lake.

As shown in Figure 3, the spatial distribution characteristics of exchangeable nitrogen in suspended solids of Lihu Lake were largely uniform. Overall, the contents in the estuary were higher than in the central lake. The estuary is constantly subjected to water disturbances, which are conducive to the adsorption of $\text{NH}_4^+\text{-N}$ and $\text{NO}_3^-\text{-N}$ onto suspended solids, resulting in high EN contents in the bay, with only slight differences between the studied zones. Nonetheless, the spatial distribution was ranked as follows: Zone D > C > B > A. Currently, a large number of submerged plants has been identified in Zone A, including *Myriophyllum verticillatum*, *Hydrilla verticillata*, and *Vallisneria spiralis*. However, other floating plants, such as *Hydrocharis dubia*, water hyacinth, and water chestnut, have been identified in other zones. Compared with floating plants, submerged plants have a relatively longer growth cycle, and their resulting detritus exists as organic nitrogen, which can only be released through mineralization. Furthermore, even after being released, it can be easily absorbed by aquatic plants to synthesize the substances that they need, which results in low nitrogen contents in the suspended solids of Zone A. Moreover, environmental dredging projects were carried out in Zones A and B to remove a large amount of pollutants from the sediment, which led to a significant reduction in nitrogen and phosphorus loads in sediments, as well as a reduction in re-suspension. Sediment dredging has also been carried out in some parts of Zone C. Additionally, Changguangxi is rich in aquatic plants, and the resulting macrophyte decay has increased the nitrogen loads in this area. Zone D is greatly affected by urban sewage, which causes a large amount of nutrients to be imported into the lake, and also increases the re-suspension of sediments. Zone D has not yet been subjected to large-scale sediment dredging, and, therefore, the nitrogen content in this zone is relatively high, resulting in a significantly higher EN content.

The EN concentration of the suspended solids of Lihu Lake ranged from 63.5 to 354.4 mg/kg (dry weight, the concentration for the solid fraction in this study is all dry weight), with a mean value of 174.4 mg/kg. The most heavily polluted were the northeastern regions of Zones A and D, whereas the areas with less pollution were mainly concentrated in the western regions of Zones A and B. Compared with other studies [37], the concentration of EN in suspended solids was higher. This could be because, after the submerged plant restoration project and fish regulation project, massive residues produced by the death of plants and fish accumulated on the surfaces of the suspended solids, resulting in high SON contents in the suspended solids. The main EN form is SON, which ranges from 17.6 to 187.9 mg/kg, with a mean value of 94.7 mg/kg, accounting for 54.3% of the EN. SON is mainly composed of free amino acids, amino sugars, urea, and low-molecular-weight organic acids, and is an important part of EN. Additionally, it plays a significant role in the mineralization, immobilization, migration, and transformation of nitrogen [38]. The organic matter (OM) in suspended solids forms a large amount of $\text{NH}_4^+\text{-N}$ via mineralization, which is then distributed between the overlying water-suspended solids and the surface sediments. Therefore, $\text{E-NH}_4^+\text{-N}$ is the most “active” component of the various forms of nitrogen in suspended solids. The maximum value of $\text{E-NH}_4^+\text{-N}$ was 218.1 mg/kg, whereas the minimum value was 10.86 mg/kg; the mean value was 62.9 mg/kg, accounting for 36.1%. Compared with other forms of nitrogen, the $\text{E-NO}_3^-\text{-N}$ content was relatively low. Specifically, its mean value was 16.8 mg/kg, accounting for only 9.6% of the EN.

3.2.2. Spatial Distribution of Acid-Hydrolyzable Nitrogen (HN) in Suspended Solids

The acid-hydrolyzable nitrogen (HN) in the suspended solids is a kind of nitrogen form that can be transformed after mineralization and then released into the overlying water. HN mainly exists in the form of organic nitrogen. The most identifiable organic compounds in HN are amino acid nitrogen (AAN), amino sugar nitrogen (ASN), ammonium nitrogen (AN), and acid-hydrolyzable unknown nitrogen-containing compounds (HUN) [39]. Figure 4 illustrates the spatial distribution of all forms of HN in suspended solids of Lake Lihu.

Figure 4. Spatial distribution of acid-hydrolyzable nitrogen (HN) in suspended solids of Lihu Lake.

The spatial distribution of various forms of HN at each sampling site was different. The mean values of AN, AAN, ASN, and HUN were 152.4, 50.6, 14.3, and 561.3 mg/kg, respectively. HUN had a lower decomposition degree than other HN forms such as AN, and belonged to the form that was not easily mineralizable. However, HUN decreased as the soil mineralization test proceeded. This indicates that HUN can also become bioavailable through biodegradation [40]. HUN had the highest proportion of HN (72.1%), followed by AN and AAN (19.6% and 6.5%, respectively). The suspended solids produce a certain amount of AN during the process of acid hydrolysis. The proportion of AN in the sampling sites of the eastern Zone A, the western Zone B, and the northern Zone D was relatively high, exceeding 28% in all cases. According to Wang et al. [41], AN is positively correlated with fixed ammonium nitrogen and organic matters. Therefore, we can infer that the organic matter and fixed ammonium nitrogen contents in suspended solids are important factors affecting AN. AAN (i.e., one of the most identifiable nitrogen-containing organic compounds), which is a kind of mineralizable nitrogen, and also a kind of stable SON [42]. Amino acids are mainly derived from proteins and polypeptides in organic matters. Thus, the OM content and its compounds will likely affect AAN contents directly. Suspended solid ASN mainly originates from the cell contents of microorganisms and aquatic insects. Its main component is glucosamine, followed by lactosamine; in fact, the sum of these two amines is close to the amount of nitrogen in the amino sugar fraction [43]. The results show that amino sugars are rarely found in higher plants [44,45], and are instead mainly concentrated in the chitin of microorganisms and insects. ASN represented the smallest fraction, with only 1.84%.

The mean values of HN in Zones A, B, C, and D were 681.9, 697.3, 814.9, and 903.5 mg/kg, respectively. Moreover, the spatial distribution was ranked as Zone D > C > B > A.

3.2.3. Spatial Distribution of Residual Nitrogen (RN) and Total Nitrogen (TN) in Suspended Solids

Figure 5 illustrates the spatial distribution of residual nitrogen (RN) and total nitrogen (TN) in suspended solids.

Figure 5. Spatial distribution of residual nitrogen (RN) and total nitrogen (TN) in suspended solids in Lihu Lake.

RN in suspended solids is mainly comprised of organic heterocyclic compounds or organic nitrogen-bound compounds with heterocyclic or aromatic rings [44]. This nitrogen fraction mainly comes from humic structural compounds with a higher degree of condensation. The RN content was found to be 189.0–1435.0 mg/kg, with a mean value of 710.3 mg/kg. The highest RN content was observed in the Changguangxi Bridge sampling site, which is in the south of Lihu Lake, followed by the Jincheng Bay sampling site in the eastern Lihu Lake. Zone A exhibited the lowest RN mean value (517.8 mg/kg). The RN spatial distribution increased from west to east (Zone D > C > B > A).

The TN content in suspended solids ranged from 758.9 to 3098.1 mg/kg, with a mean value of 1663.2 mg/kg. Moreover, the spatial distribution of TN content in suspended solids varied greatly. The TN content in the eastern Zone A was the highest, exhibiting concentrations that were 1.86 times higher than the mean value of the entire lake. The mean values of the four zones (A, B, C, D) were 1380.2, 1403.6, 1663.0, and 2168.9 mg/kg, respectively.

The average proportions of various nitrogen forms in each zone are illustrated in Figure 6. Based on the proportion of the content of each nitrogen form in the TN, HN had the highest proportion, accounting for 28.4–60.8%, with an average of 48.4%, followed by RN, accounting for 22.2–55.0%, with an average of 40.55%. EN had the smallest proportion, accounting for only 11.03%. The content of EN in Area D was relatively high, which was likely due to the high TN content in the overlying water of Zone D. This conclusion was based on the Surface Water Environment Quality Standard of China, which classified the aforementioned area as Class IV–V. The suspended solids adsorb inorganic nitrogen (e.g., ammonia nitrogen and nitrate nitrogen) onto the surfaces of the particles via exchangeable adsorption.

Figure 6. Contents of various nitrogen forms in each zone.

3.3. Spatial Distribution of Phosphorus Forms in Suspended Solids

3.3.1. Spatial Distribution of Inorganic Phosphorus in Suspended Solids

The spatial distribution of weakly adsorbed inorganic phosphorus (WA-P_i), potentially active inorganic phosphorus (PA-P_i), iron (Fe)- or aluminum (Al)-bound inorganic phosphorus (Fe/Al-P_i), and calcium (Ca)-bound inorganic phosphorus (Ca-P_i) is shown in Figure 7. As illustrated in Figure 7, the inorganic phosphorus contents in the suspended solids of Lihu Lake exhibited overall lower concentrations in the west than in the east, and the highest values were mostly concentrated in Zone D.

Figure 7. Spatial distribution of inorganic phosphorus in suspended solids of Lihu Lake.

WA-P_i is mainly derived from interstitial water or inorganic phosphorus types that adhere to other phases, such as carbonate, oxide, hydroxide, or clay mineral particles, in a physically adsorbed state, and is thus a form of phosphorus that can be easily transported [46,47]. The WA-P_i content in suspended solids of Lihu Lake ranges from 0.6 to 24.7 mg/kg, with a mean value of 10.3 mg/kg. Notably, its fraction only accounts for 2.7% of the total inorganic phosphorus and 1.6% of the total phosphorus. The highest values of WA-P_i occurred in the central region of Zone A and Zone D, whereas the lowest values were mainly concentrated in the western part of Zone A and Zone B in western Lihu Lake.

PA-P_i in suspended solids refers to the fraction of active Fe-P and Al-P salts leached by a NaHCO₃ solution during hydrolysis. PA-P_i can be used as an effective indicator of the bioavailable phosphorus content in suspended solids [48]. In this study, PA-P_i content in the suspended solids ranged from 19.3 to 128.6 mg/kg, with an arithmetic mean value of 75.2 mg/kg. This proportion accounted for 19.68% of the inorganic phosphorus and 11.71% of total phosphorus.

Fe/Al-P_i refers to the inorganic phosphorus extracted by a NaOH solution, and includes phosphorus combined with Fe/Al oxides or hydroxides. When the suspended solid overlying water interface is under anaerobic conditions, Fe/Al-P_i can be converted into soluble phosphorus, which is then recycled into the water column [49]. The Fe/Al-P_i content was 21.9–236.1 mg/kg, with a mean value of 111.1 mg/kg.

Ca-P_i refers to the phosphorus fraction in the sediments that is associated with phosphorus-bearing minerals, such as authigenic apatite, calcium carbonate deposits, or biological skeletons [50]. Notably, this fraction is known to be released in acidic environments. However, the pH of the lake's suspended solids is generally weakly alkaline, and thus has little effect on the release of Ca-P_i from the suspended solids to the water column. Therefore, Ca-P_i in the suspended solids is not readily bioavailable to aquatic organisms. Ca-P_i contents were found to range from 93.9 to 307.8 mg/kg, with a mean value of 185.4 mg/kg.

3.3.2. Spatial Distribution of Organic Phosphorus in Suspended Solids

Figure 8 illustrates the spatial distribution of organic phosphorus in suspended solids. Similarly to WA-P_i, weakly absorbed organic phosphorus (WA-P_o) is also derived from interstitial water or an organic phosphorus form that adheres to other phases, such as carbonate, oxide, hydroxide, and clay ore particles, by physical adsorption. Additionally, it is a form that is extremely easy to mineralize and transfer [51]. In this study, the content of WA-P_o in suspended solids of Lihu Lake ranged from 1.9 to 11.0 mg/kg, with an average value of 4.7 mg/kg.

The potentially active organic phosphorus (PA-P_o) fraction is mainly comprised of phosphorus-containing compounds, such as nucleic acids, phospholipids, and phosphosaccharides extracted from NaHCO₃ solutions. [52,53]. Moreover, it can be used as an effective phosphorus source for the growth of phytoplankton and aquatic organisms. The PA-P_o content in suspended solids of Lihu Lake ranged from 9.2 to 45.2 mg/kg, with an average value of 24.4 mg/kg.

Medium active organic phosphorus (MA-P_o) is the potentially bioavailable phosphorus form in suspended solids, which is composed of medium active organic phosphorus extracted by a NaOH solution (MA_{NaOH-Po}) and by a hydrochloric acid solution (MA_{HCl-Po}), including calcium phytate, magnesium phytate, and some phosphorus-containing compounds combined with fulvic acids. These compounds can be further decomposed into inorganic phosphorus through the action of hydrolases, such as phosphatase and phytase [54]. The average MA-P_o value was 65.3 mg/kg, ranging from 36.3 to 103.4 mg/kg.

Figure 8. Spatial distribution of organic phosphorus in suspended solids of Lihu Lake.

Non-active organic phosphorus (NA-P_o) is a relatively stable phosphorus form in suspended solids, which is composed of non-active organic phosphorus extracted by NaOH solutions (NA_{NaOH}-P_o) and residual phosphorus (R-P_o), including humic acid in humus, organic phosphorus in humin, iron-aluminum salts containing 4–6 kinds of phosphoric acids in inositol, and a small amount of phosphorus-containing aluminum or calcium encapsulated by an Fe₂O₃ adhesive film [55]. In this study, the range of NA-P_o in suspended solids was 134.0–274.1 mg/kg, and the average value was 172.3 mg/kg, accounting for 67.7% of the total organic phosphorus and 25.1% of the total phosphorus. Additionally, the results demonstrated that the majority of NA-P_o was R-P_o, and the proportion of R-P_o in NA-P_o ranged from 64.9% to 91.1%, with an average of 80.4%.

The total phosphorus contents of suspended solids in Lihu Lake were similar to those in the Taihu Lake basin [56]; when compared with the sediment in Taihu Lake [57], the contents were significantly higher.

The proportions of various phosphorus forms in each zone are illustrated in Figure 9. In this study, inorganic phosphorus was the primary form of phosphorus in suspended solids, with contents ranging from 144.1 to 684.5 mg/kg, and an average of 381.9 mg/kg, accounting for 58.6% of the total phosphorus. Inorganic phosphorus was dominated by Ca-P_i, accounting for 49.6% of the inorganic phosphorus fraction. Furthermore, organic phosphorus was mainly composed of R-P_o in inactive organic phosphorus, also accounting for more than 50% of the total organic phosphorus, which is consistent with previous studies [56]. Organic phosphorus (OP) mainly derives from terrestrial sources

and biological processes, and OP content is generally regarded as a better eutrophication indicator than TP. In this study, the contents of inorganic and organic phosphorus with direct bioavailability ranged between 22.0–153.3 and 12.6–51.2 mg/kg, respectively, accounting for 7.5–18.3% and 1.5–7.4%. The content of organic phosphorus with direct bioavailability was relatively low, which may be due to the strong interaction between the suspended solids and water in the lake. This facilitates the decomposition of WA-P_o and PA-P_o into small-molecule compounds or phosphate, which are then released into the overlying water.

Figure 9. Contents of inorganic (a), organic (b), and bioavailable (c) phosphorus forms in each zone.

In nature, organic phosphorus occurs mainly in the form of monoester or diester phosphorus. Previous studies showed that hydroxyl-bound phosphorus in monoester and diester organic phosphorus has similar properties to those of orthophosphate, both of which absorb and desorb onto particles via hydroxyl-phosphorus interactions [58]. Furthermore, organic phosphorus shares similar adsorption and desorption mechanisms with inorganic phosphorus in suspended solids. In fact, phosphorus is always in a state of dynamic equilibrium between the suspended solids and overlying water. They can transform each other under certain conditions, and some forms of phosphorus in suspended solids can be released into the overlying water through physical, chemical, and biological processes, thereby adopting forms that can be readily utilized by organisms. In this study, we first observed that WA-P_i, WA-P_o, PA-P_i, and PA-P_o were bioavailable phosphorus compounds that could be released into overlying water and participate in water recycling, which affects the nutritional status and primary productivity of the water body. Secondly, Fe/Al-P_i and MA-P_o can be converted under certain conditions. Specifically, Fe/Al-P_i can be released to meet the needs of plankton growth when the suspended solid–water interface presents anaerobic conditions, whereas MA-P_o can directly generate inorganic phosphorus under the action of hydrolases, such as phosphatase and phytase, or otherwise be mineralized to soluble active phosphorus via microorganism activity. Generally, Fe/Al-P_i and MA-P_o are regarded as potentially bioavailable phosphorus forms [59,60]. Lastly, Ca-P_i and NA-P_o are considered to be stable forms, and therefore cannot be easily converted into bioavailable phosphorus forms, especially in lakes that are already affected by eutrophication. Therefore, the bioavailability of various forms of phosphorus in suspended solids must be further studied via multiple methods, such as mineralization and enzymatic hydrolysis, in order to establish a better understanding of nutrient dynamics in eutrophic lakes.

3.4. Correlation Analysis Indicative of Nutrient Sources

Suspended solids in lakes originate from numerous sources, and the correlation between various forms of nitrogen and phosphorus can reflect the link between each fraction. Furthermore, this approach can reflect the sources of nitrogen and phosphorus forms in suspended solids to some extent. In order to explore the possible sources of nitrogen and phosphorus forms in suspended solids, Pearson correlation tests were conducted between all fractions. The analysis results are summarized in Table 1; Table 2.

Table 1. Correlations between nitrogen forms in suspended solids.

	ETN	E-NH ₄ ⁺ -N	E-NO ₃ ⁻ -N	SON	HN	AN	AAN	ASN	HUN	RN	TN
ETN	1										
E-NH ₄ ⁺ -N	0.794 **	1									
E-NO ₃ ⁻ -N	0.736 **	0.549 **	1								
SON	0.539 **	−0.078	0.359	1							
HN	0.440 *	0.198	0.759 **	0.365	1						
AN	0.577 **	0.403 *	0.745 **	0.325	0.854 **	1					
AAN	0.178	0.374	−0.004	−0.201	−0.216	−0.050	1				
ASN	−0.045	0.030	−0.039	−0.115	0.218	0.234	0.301	1			
HUN	0.265	−0.014	0.630 **	0.371	0.929 **	0.659 **	−0.517 **	0.073	1		
RN	0.643 **	0.558 **	0.814 **	0.216	0.769 **	0.816 **	0.072	0.089	0.593 **	1	
TN	0.656 **	0.481 **	0.862 **	0.338	0.911 **	0.888 **	−0.036	0.140	0.764 **	0.959 **	1

In Table 1, the numbers represent the mean correlation coefficients; ** means $p < 0.01$, and indicates an extremely significant correlation; * means $p < 0.05$, and indicates a significant correlation. represents an extremely significant positive correlation; represents a significant positive correlation; represents a positive correlation; represents an extremely significant negative correlation; represents a negative correlation.

Table 2. Correlations between phosphorus forms in suspended solids.

	WA-P _i	PA-P _i	Fe/Al-P _i	Ca-P _i	WA-P _o	PA-P _o	MA-P _o	NA-P _o	IP	OP	TP
WA-P _i	1										
PA-P _i	0.910 **	1									
Fe/Al-P _i	0.728 **	0.817 **	1								
Ca-P _i	0.670 **	0.616 **	0.626 **	1							
WA-P _o	0.344	0.234	0.201	0.142	1						
PA-P _o	0.256	0.293	0.374	0.332	0.209	1					
MA-P _o	0.346	0.492 *	0.563 **	0.419 *	0.294	0.159	1				
NA-P _o	0.568 **	0.634 **	0.822 **	0.467 *	0.181	0.377 *	0.414 *	1			
IP	0.844 **	0.868 **	0.899 **	0.887 **	0.212	0.380 *	0.529 **	0.707 **	1		
OP	0.588 **	0.663 **	0.856 **	0.530 **	0.285	0.499 **	0.612 **	0.960 **	0.757 **	1	
TP	0.805 **	0.848 **	0.936 **	0.816 **	0.249	0.443 *	0.588 **	0.834 **	0.975 **	0.884 **	1

In Table 2, the numbers represent the mean correlation coefficients; ** means $p < 0.01$, and indicates an extremely significant correlation; * means $p < 0.05$, and indicates a significant correlation. represents an extremely significant positive correlation; represents a significant positive correlation; represents a positive correlation.

The correlation analysis results showed that TN was significantly positively correlated with EN, HN, and RN ($p < 0.01$, $n = 28$). Among these, the correlation coefficient between TN and RN was the highest ($r = 0.959$), indicating that the TN content in suspended solids was mainly modulated by RN.

E-TN is significantly positively correlated with $\text{E-NH}_4^+\text{-N}$, $\text{E-NO}_3^-\text{-N}$, and E-SON ($p < 0.01$, $n = 28$). Among these, the correlation coefficient of E-TN and $\text{E-NH}_4^+\text{-N}$ was the highest ($r = 0.794$). Additionally, $\text{E-NH}_4^+\text{-N}$ was one of the main components of E-TN, and thus, it can be inferred that E-TN is mainly controlled by $\text{E-NH}_4^+\text{-N}$. Among the different nitrogen forms, $\text{E-NH}_4^+\text{-N}$ was significantly positively correlated with $\text{E-NO}_3^-\text{-N}$ ($r = 0.549$, $p < 0.01$, $n = 28$), but not significantly positively correlated with E-SON. This indicates that $\text{E-NH}_4^+\text{-N}$ and $\text{E-NO}_3^-\text{-N}$ can transform into each other under certain conditions, mainly because of the DO (Dissolved Oxygen) content in Lihu Lake ($\text{DO} = 7.05$), which facilitates denitrification. Moreover, there was no correlation between E-SON, $\text{E-NH}_4^+\text{-N}$, and $\text{E-NO}_3^-\text{-N}$. This may be because the transformation between inorganic and organic nitrogen occurs mainly through anaerobic microorganism-mediated mineralization; however, the conditions in the overlying water were not favorable for the growth of anaerobic microorganisms. Furthermore, H-TN was significantly positively correlated with AN and HUN ($r = 0.854$, $r = 0.929$, $p < 0.01$, $n = 28$), meaning that the amount of H-TN in suspended solids mainly depended on AN and HUN. Among the different nitrogen forms, only AN and HUN had a positive correlation ($r = 0.659$, $p < 0.01$, $n = 28$), indicating that the two forms may not only share a similar source, but frequent transformations likely occur between them. The correlations among the remaining forms were not significant, indicating that they may derive from different sources. Particularly, AAN and HUN showed a significant negative correlation ($r = -0.517$, $p < 0.01$, $n = 28$), suggesting that AAN and HUN may be antagonistic to each other or derive from different sources.

To explore the potential sources of phosphorus in suspended solids, a correlation analysis among the phosphorus forms was performed. There was a significant positive correlation between TP and inorganic phosphorus, as well as MA-P_o and NA-P_o ($p < 0.01$), meaning that the TP content was mainly affected by inorganic phosphorus, MA-P_o , and NA-P_o . Moreover, the correlation between TP and various forms of inorganic phosphorus was more significant, indicating that TP in suspended solids is mainly controlled by inorganic phosphorus. In other words, the various forms of inorganic phosphorus originate from sediment re-suspension. Furthermore, IP, MA-P_o , and NA-P_o were significantly positively correlated ($p < 0.01$), indicating that they may share the same source. Particularly, the correlation between WA-P_i and PA-P_i with high biological activity was the most significant ($r = 0.910$, $p < 0.01$, $n = 28$), suggesting that these two forms likely share the same source. We also observed a significant correlation between OP and MA-P_o , as well as MA-P_o and NA-P_o ($p < 0.01$). However, WA-P_o did not have a significant correlation with the contents of other phosphorus forms. This may be because a fraction of OP is bioavailable phosphorus, which mainly comes from agricultural non-point source pollution [61]. However, NA-P_o mainly derives from insoluble phosphorus minerals, and, therefore, cannot be easily utilized by aquatic organisms. Based on these observations, we concluded that the sources of OP in suspended solids were quite variable in the studied lake.

Previous studies [62,63] showed that the dry and wet deposition amount of TN was 11.0 t/a, whereas that of TP was 0.2 t/a. Moreover, the release fluxes of nitrogen and phosphorus in sediments by resuspension were approximately 20–30 t/a and 1–2 t/a, respectively. The sum of the two accounted for more than 90% of the nitrogen and phosphorus loads in Lihu Lake. Dry and wet nitrogen deposition is mainly attributable to dissolved total nitrogen (DTN) (91.4%), whereas the contribution of dissolved total phosphorus (DTP) to phosphorus deposition is relatively lower, with an average of 65.1%. This is also consistent with the fact that TN was found to be mainly dissolved nitrogen, whereas TP was largely comprised of the particulates in the water of Lihu Lake. These observations also reflect the contribution of suspended solids to nitrogen and phosphorus pollution in Lihu Lake. The prevention and control of endogenous pollution in lakes is a complicated and difficult undertaking. Internal loading and eutrophication affect each other. Therefore, to improve the water quality of Lihu Lake, it is necessary

to consider not only the interaction between water and sediments, but also the impact of suspended solids on pollution loads.

3.5. Analysis of Influencing Factors

Water is the medium that connects the various environmental components of a lake [64]. Therefore, the characteristics of the overlying water must have an important effect on the release of nitrogen and phosphorus in suspended solids. In order to further characterize the relationship between the overlying water, suspended solids, and surface sediments, Pearson's correlation analyses were conducted between physical and chemical indices and the nitrogen and phosphorus forms in suspended solids and surface sediments. The results are illustrated in Figure 10.

Figure 10. Relationships between nitrogen and phosphorus forms in suspended solids and surface sediments and physicochemical indices in water. The numbers in the figure indicate p values. (a) correlation between nitrogen forms in suspended solids and water, (b) correlation between nitrogen forms in surface sediments and water, (c) correlation between phosphorus forms in suspended solids and water, (d) correlation between phosphorus forms in surface sediments and water. * means $p < 0.05$, ** means $p < 0.01$, *** means $p < 0.001$. Red cells indicate positive correlations and blue cells indicate negative correlations.

Although suspended solids have a strong capacity to release EN and HN, they exhibited no significant correlation with TN in overlying water. In contrast, RN in suspended solids showed a positive correlation with TN, DTN, and ammonium nitrogen in the overlying water. This was mainly

because the EN and HN contents were less than that of RN, which also demonstrates that RN was the main nitrogen source in the overlying water. The content of bioavailable N forms in suspended solids is positively correlated with NH_4^+ -N in the water, which means that under certain conditions, the suspended solids can affect the content of ammonia in water. TN and NH_4^+ -N in the water had a significantly positive correlation with EN and E- NH_4^+ -N in the surface sediments ($p < 0.01$), and this was consistent with Sun's [47] observations in Erhai Lake. EN is a nitrogen form that is easily released into the overlying water if the conditions are favorable. Therefore, this relationship indicated that the nitrogen in the overlying water mainly originated from the surface sediments, and the release of NH_4^+ -N from surface sediments played an important role in the supply of NH_4^+ -N to the overlying water. Both NH_4^+ -N and NO_3^- -N in the surface sediment had significant correlations with HN. This indicates that the mineralization and decomposition of HN in the surface sediments is an important supplier to these two nitrogen forms, and can indirectly provide a continuous nitrogen source for overlying water. In suspended solids and surface sediments, various forms of nitrogen were mainly significantly linked to ammonium nitrogen in the overlying water, indicating that both of them contribute to the reduction of nitrogen in water to ammonia nitrogen. Some studies [65,66] have shown that algae can selectively utilize nitrogen in water. This is because ammonium nitrogen does not need to be reduced when assimilated, whereas nitrate nitrogen needs to be reduced before its use. Therefore, ammonia nitrogen promotes the growth of algae more effectively. The inorganic nitrogen in water is mainly composed of ammonia nitrogen, which is not correlated with the concentration of suspended solids. Therefore, although the RN content in suspended solids is significantly positively correlated with ammonia nitrogen ($p < 0.01$), RN cannot be directly converted to ammonia nitrogen and, consequently, suspended solids cannot directly promote algae growth. Therefore, we can infer that the sediment has a greater effect on the nitrogen in water compared with suspended solids. In contrast, high concentrations of suspended solids lead to water turbidity, thereby limiting algae photosynthesis and inhibiting their growth to some extent.

Chlorophyll a (*Chl.a*) is an important indicator of primary productivity. In this study, the correlation coefficients between *Chl.a* and various forms of phosphorus in suspended solids were all found to be positive. Among them, the correlation coefficient between *Chl.a* and inorganic phosphorus was the highest ($r = 0.567$). In other words, when the external environment changes, part of the inorganic phosphorus fraction is released, thereby increasing the content of phosphorus in water. Algae are parts of the suspended solids; in the meanwhile, phosphorus in suspended solids plays an important role in algae growth. Particularly, its correlation coefficient with Ca- P_i was the highest ($r = 0.616$). This is because areas with high *Chl.a* contents are indicative of high photosynthetic activity and active phytoplankton growth, which translates to an accelerated rate of biological residue decomposition and a subsequent increase in Ca- P_i that result from the combination of biological residues with calcium in the environment. The bioavailable phosphorus forms in the sediment can be released into the water, and then affect the algae growth. All of the nitrogen forms in suspended solids and sediment are not correlated with *Chl.a*, which means that nitrogen in suspended solids and sediment does not directly affect the primary productivity of the lake.

The correlation between the phosphorus components in the water and phosphorus forms in the suspended solids/surface sediments demonstrated that the content of phosphorus in water was affected by both suspended solids and sediment. $\rho(\text{TP})$ in the overlying water was affected by various phosphorus forms in the suspended solids, especially inorganic phosphorus. Notably, particulate phosphorus (PP) was found to be the main component of TP in the overlying water, and $\rho(\text{PP})$ was mainly affected by inorganic phosphorus in the suspended solids. Moreover, the correlation coefficient of Fe/Al- P_i was the highest ($r = 0.503$), indicating that Fe/Al- P_i was the main form affecting TP content in the water. The increase of $\omega(\text{PA-}\text{P}_i)$ in the surface sediments leads to increases in $\rho(\text{IP})$ in the overlying water. This observation is highly relevant, as IP is known to be easily utilized by algae, which leads to cyanobacterial blooms. The bioavailable phosphorus forms in sediment affect the nutrient level by affecting the content of IP in water.

3.6. Comparison Analysis between Suspended Solids and Surface Sediments

Nutrients in the sediments can be released into the overlying water through diffusion, convection, and other processes, causing secondary pollution [67]. Moreover, suspended solids are an important part of aquatic ecosystems, and their nutrient concentrations can reflect the water quality of a lake [68]. In order to more precisely control the endogenous pollution of a lake, it is necessary to compare the contents of various nutrient forms in suspended solids with those in surface sediments. These results are illustrated in Figure 11.

Figure 11. Comparison between the contents of nitrogen and phosphorus forms in the suspended solids with those in surface sediments.

Liu [69] examined the correlation between the content of various nitrogen forms and particle size in sediments of the Dan River. The results showed that finer sediment particles translated to higher contents of nitrogen forms. Therefore, the nitrogen adsorption capacity of suspended solids is stronger than that of surface sediments. Moreover, EN is the main and most active form involved in the transportation and migration of nitrogen in suspended solids/surface sediments. It is also the main source of bioavailable nitrogen in suspended solids and surface sediments, and the NH₄⁺-N and NO₃⁻-N content in suspended solids is typically lower than in surface sediments. This is because the nitrogen concentration in the interstitial water of surface sediments is higher than that in the overlying water due to the influence of OM degradation by benthic organisms. Therefore, suspended solids adsorb less NH₄⁺-N and NO₃⁻-N than surface sediments. Moreover, low O₂ concentrations in sediments favor the growth of denitrifying bacteria, thereby promoting the denitrification process and the subsequent accumulation of large amounts of NH₄⁺-N in the surface sediments. Thus, the NH₄⁺-N content was higher than that of NO₃⁻-N. The OM content affects the number of adsorptive sites in sediments. Studies have shown that sediments with high organic matter contents have more adsorptive sites, which can adsorb more AN and AAN. In consequence, organic matter affects AN and AAN contents. The COD (Chemical Oxygen Demand) content in the overlying water of Lihu Lake (COD = 4.41 mg/L) is lower than that of the interstitial water. Therefore, the contents of AN and AAN in the suspended solids are lower than those of surface sediments. Generally speaking, there are three main sources of bioavailable nitrogen in suspended solids/surface sediments: Inorganic nitrogen (NH₄⁺-N, NO₃⁻-N), dissolved organic nitrogen, and mineralizable organic nitrogen (mainly AAN). In this study, EN, AN, and AAN occurred as bioavailable

nitrogen, and their contents in suspended solids were lower than those in surface sediments. In other words, the nitrogen in surface sediments was more bioavailable than that in suspended solids.

Upon comparing the RN content in the suspended solids with that in the surface sediments, we observed that the RN content in suspended solids of each sampling site was generally more than twice that of the surface sediments. This was because the oxidation environment of the suspended solids was likely stronger than that of surface sediments, and a strong oxidation environment enhances the stability of RN in sediments [70].

In general, the contents of various forms of phosphorus except for MA-P_o in suspended solids were higher than in the surface sediments. That is, the phosphorus in suspended solids was more bioavailable than that in surface sediments. This relationship between suspended solids and surface sediments was consistent with the observations of Jin [37] and Zan [61]. This was mainly attributed to the continuous wind–wave disturbances in the studied sites, which cause the surface sediments to be constantly disturbed. For this reason, only low-density substances (including phytoplankton, zooplankton, organic detritus, algae fragments, and relatively light minerals) remain in the water column for long periods.

Higher WA-P contents in suspended solids indicate that the adsorption of inorganic phosphate and dissolved organic phosphorus onto suspended solids was higher than that of the surface sediments. Compared with surface sediments, suspended solid particles are smaller and their specific surface area increases when they enter the water body after being disturbed. Therefore, this increase in surface potential energy facilitates adsorption. This is consistent with the results of a simulation test, which demonstrated that the amount of adsorption and desorption of phosphate increased with decreased particle sizes. Previous studies [71] have shown that the content of inorganic phosphate and dissolved organic phosphorus in interstitial water was much higher than that in overlying water, which results in the diffusion and release of dissolved phosphorus from the interstitial water to the overlying water. According to a field investigation in Lake Arresø, a shallow lake in Denmark [72], the increase of nutrients released from dynamic suspension can reach up to 20–30 times the original value. This kind of release is likely aggravated by wave and wind disturbances, even though the content of dissolved phosphorus in the lake remains stable. Therefore, given that a large amount of released dissolved phosphorus can be re-adsorbed onto the suspended solids, the phosphorus content dissolved in the water likely remains relatively stable. This conclusion also supports the phosphate buffering mechanism proposed by Froelich [73]. In a few words, this rapid adsorption is beneficial to maintaining the balance of phosphorus in the water. Nevertheless, the suspended solids can also adsorb the dissolved phosphorus from the surface sediments, which is then released under certain conditions.

The NA-P_o content in suspended solids was significantly higher than that of the surface sediments, which may be due to the large amount of loose and amorphous inorganic macromolecular flocculate materials or organic detritus from surface sediment entering the water, increasing the organic phosphorus content in water. These substances not only have poor precipitation themselves, but also lead to poor precipitation of suspended solids after being adsorbed, which further leads to significant increases in the NA-P_o content of suspended solids.

According to the correlation analysis, the dissolved phosphorus content in the water of Lihu Lake is affected by both suspended solids and sediment. However, since high phosphorus contents are found in suspended solids, we should pay more attention to the suspended solids.

4. Conclusions

This study investigated the contents, spatial distribution, and sources of nitrogen and phosphorus forms in the suspended solids of Lihu Lake, and comparatively analyzed the different effects of suspended solids and surface sediments on water quality. After a comprehensive analysis and discussion of the results, our main conclusions were the following:

- (1) The content of TN in the suspended solids of Lihu Lake was 758.9–3098.1 mg/kg and showed a decreasing trend from east to west. The proportions of various N forms in the suspended solids

of the study areas were ranked as follows: Hydrolyzable nitrogen (HN) > residual nitrogen (RN) > exchangeable nitrogen (EN). The total phosphorus (TP) ranged from 294.8 to 1066.4 mg/kg, the contents of IP and OP in suspended solids were 381.9 and 259.8 mg/kg, respectively, and Ca-Pi and NA-P_o were the major components of IP and OP. Almost all P forms exhibited higher contents in the west than in the east. This is because several environmental dredging and ecological restoration projects have been carried out in western Lihu Lake.

- (2) The correlation analysis of various forms of nitrogen and phosphorus in suspended solids indicated that there were different sources of suspended nitrogen for Lihu Lake, and TN in suspended solids was mainly affected by RN. Inorganic phosphorus in suspended solids likely originated from a common source, whereas organic phosphorus came from different sources. Phosphorus in suspended solids was controlled by both IP and OP, but was mainly affected by IP.
- (3) The nitrogen in sediments was more bioavailable than that in suspended solids. In particular, once the physical and biochemical conditions of the sediment change, the EN in the sediment can be released and can diffuse into the overlying water, affecting the contents of NH₄⁺-N and TN in water. The nitrogen forms in SS change continuously through rapid transformations when water quality conditions change. The contents of phosphorus in water were affected both by suspended solids and sediment. The bioavailable phosphorus content in suspended solids was higher than that in sediments, and the suspended solids influenced the $\rho(\text{TP})$ by affecting particulate phosphorus. Suspended solids act as a medium that continually carries phosphorus from sediments to water and provides a substrate that enables phosphorus to remain in the water column for long periods.

Although many studies have been conducted on the nutrient concentrations in water and sediment, little is known about the nutrients in suspended solids. The spatial distribution characteristics of nitrogen and phosphorus forms in suspended solids were analyzed in this study. The impact on eutrophication was studied based on statistical analysis of these data. Future studies will hopefully be on the biogeochemical mechanisms, so as to provide theoretical support for controlling the eutrophication.

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Appendix A

Table A1. The abbreviations and their meanings in this study.

Abbreviation	Meaning
SS	suspended solids
TSS	total suspended solids
OM	organic matter
TN	total nitrogen
DTN	dissolved total nitrogen
PN	particulate nitrogen
TP	total phosphorus
DTP	dissolved total phosphorus
PP	particulate phosphorus
IP	inorganic phosphorus
OP	organic phosphorus
EN	exchangeable nitrogen

Table A1. Cont.

Abbreviation	Meaning
E-NH ₄ ⁺ -N	exchangeable ammonium nitrogen
E-NO ₃ ⁻ -N	exchangeable nitrate nitrogen
SON	exchangeable soluble organic nitrogen
HN	acid-hydrolyzable nitrogen
AAN	amino acid nitrogen
ASN	amino sugar nitrogen
AN	acid-hydrolyzable ammonium nitrogen
HUN	acid-hydrolyzable unknown nitrogen
RN	residual nitrogen
WA-P _i	weakly adsorbed inorganic phosphorus
PA-P _i	potentially active inorganic phosphorus
Fe/Al-P _i	iron (Fe)- or aluminum (Al)-bound inorganic phosphorus
Ca-P _i	calcium (Ca)-bound inorganic phosphorus
WA-P _o	weakly absorbed organic phosphorus
PA-P _o	potentially active organic phosphorus
MA-P _o	medium active organic phosphorus
NA-P _o	non-active organic phosphorus
R-P _o	residual phosphorus

Figure A1. Measurement procedure for nitrogen fractionation in the suspended solids.

Table A2. Determination methods for nitrogen forms in suspended solids.

Index	Determination Method	Reference
EN, HN	Alkaline potassium persulfate digestion UV spectrophotometric method	HJ 636-2012 from Ministry of Ecology and Environment, PRC
E-NH ₄ ⁺ -N, AN	Nessler's reagent spectrophotometry	HJ 535-2009 from Ministry of Ecology and Environment, PRC
E-NO ₃ NO ₃ ⁻ -N	Ultraviolet spectrophotometry	HJ/T 346-2007 from Ministry of Ecology and Environment, PRC
SON	SON = EN - E-NH ₄ ⁺ -N - E-NO ₃ ⁻ -N	
AAN	Ninhydrin colorimetric	1. Shao J.L.; Li Q.W.; Dong B.S.; Liu H.S. Determination of total free amino acid in tea by Ninhydrin colorimetry. <i>China Food Additives</i> 2008 , 02, 162–165.

Table A2. Cont.

Index	Determination Method	Reference
ASN	Elson–Morgen colorimetric method	Bremner J.M. Organic forms of nitrogen. Madison: American Society of Agronomy, 1965: 1148–1178.
HUN	HUN = TH-AN-AAN-ASN	
RN, TN	Modified Kjeldahl method	HJ 717 - 2014 from Ministry of Ecology and Environment, PRC

Figure A2. Measurement procedure for phosphorus fractionation in the suspended solids.

Table A3. Determination methods for phosphorus forms in suspended solids.

Index	Measurement	Reference
inorganic phosphorus	phosphomolybdenum blue spectrophotometric method	HJ 593-2010 from Ministry of Ecology and Environment, PRC
TP	ammonium molybdate spectrophotometric method	GB 11893-89 from Ministry of Ecology and Environment, PRC
organic phosphorus	OP = TP – IP

Figure A3. Relationship between annual mean total suspended solids (TSS), nitrogen, and phosphorus in the water of Lihu Lake.

Table A4. Basic information of water quality of Lihu Lake.

Index	Unit	Min	Max	Average	SD ¹
T	°C	18.50	19.30	18.88	0.22
pH	/	7.84	8.37	8.15	0.09
DO	mg/L	3.60	8.50	7.05	0.96
ORP	mV	166.90	247.20	197.72	22.59
COD	mg/L	3.42	8.06	4.41	0.82
Chl.a	mg/m ³	6.30	50.54	16.20	11.11
TSS	mg/L	4	47	27	13.69
TN	mg/L	0.26	0.90	0.53	0.20
DTN	mg/L	0.04	0.41	0.18	0.11
NH ₄ ⁺ -N	mg/L	0.03	0.25	0.11	0.05
NO ₃ ⁻ -N	mg/L	0.008	0.069	0.048	0.007
TP	mg/L	0.05	0.23	0.12	0.04
DTP	mg/L	0.004	0.114	0.032	0.031
IP	mg/L	0.0006	0.0205	0.0087	0.006

¹ standard deviation.

Table A5. General characteristics of nitrogen forms in surface sediments of Lihu Lake (content unit: mg/kg).

Index	Min	Max	Average	SD
ETN	33.42	431.19	144.50	95.27
E-NH ₄ ⁺ -N	3.12	222.66	75.31	56.92
E-NO ₃ ⁻ -N	4.99	85.45	21.59	15.89
SON	17.76	126.22	47.60	30.42
HN	477.91	1156.33	701.96	158.99
AN	208.28	698.38	335.19	108.17
AAN	10.10	383.07	162.91	94.27
ASN	3.49	29.17	11.79	5.81
HUN	62.29	281.15	192.08	61.17
RN	0	1163.30	451.17	315.24
TN	516.34	2551.14	1297.64	497.75

Table A6. General characteristics of phosphorus forms in surface sediments of Lihu Lake (content unit: mg/kg).

Index	Min	Max	Average	SD
WA-P _i	1.40	20.50	6.37	4.50
PA-P _i	10.34	107.15	53.85	24.84
Fe/Al-P _i	40.93	243.13	106.93	51.85
Ca-P _i	62.10	348.53	163.43	80.71
WA-P _o	0.67	10.69	3.01	1.92
PA-P _o	0.03	54.00	9.52	10.11
MA-P _o	50.97	142.15	91.29	18.85
NA-P _o	76.93	244.99	137.07	45.83

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