Water and sediment regulation eluting and washland planting lead to nitrogen increase in the lower reaches of the Yellow River

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Abstract: The Yellow River, as an important agricultural production base in China, plays a key role in the terrestrial-sea transport and transformation of nitrogen (N). However, the reason for the transient increase in N concentration in the lower reaches of the Yellow River (LYR) remains unclarified. In this work, the contributions to the transient N elevation in the LYR of water column, suspended particulate matter, surface sediments in the LYR and the washland soils along LYR were explored throughout the Water and sediment regulation (WSR) event in 2023, respectively. The results showed that average dissolved nitrate concentrations in LYR before and during WSR were 1.38 and 1.12 times higher than those after WSR, respectively. This was primarily due to the WSR eluting the excess N slowly leached from the original beach, resulting in a decrease in N concentration after WSR. Meanwhile, redundancy analyses of water environmental factors and N and phosphorus concentrations showed that suspended particulate matter concentrations had essentially no effect on dissolved nitrate in the water column. In addition, the risk of N release was low in suspended particulate matter and surface sediments (ion exchangeable form N content ranged from 0.007 to 0.033

mg·g^{-1}), while it was high in soil (average ion exchangeable form N content was 0.092 mg·g^{-1}). Soil site with high reactive N (S-13) was a source of nitrate to the LYR. The results of leaching of nitrate by suspended particulate matter and surface sediments showed that the nitrate concentration in the water column was not significantly affected by the reduction of suspended particulate matter. In contrast, in soil S-13, the estimated leaching rate of nitrate averaged 14.74%, and ion exchangeable form N accounted for 19.25% of total N, with 76.56% of ion exchangeable form N leached. Therefore, the WSR eluting and continuous leaching of N from washland soils around the LYR notably increase the N concentration in LYR.

Key words: Soil; Nitrogen cycle; Yellow River; Washland planting; Water and sediment regulation event

1. Introduction

Nitrogen (N) is a fundamental biogenic element for the growth of plants, animals and humans. N has brought unlimited benefits to mankind when it comes to increasing food production (Duan et al., 2024). Studies show that the increase in N fertilizer consumption and low N use efficiency (only 25%) for crop production, resulting in large amounts of reactive N being released from the soil into the environment, especially the aqueous environment (Wang et al., 2024; Zhang et al., 2015). In global N cycle, rivers are key carriers for N transport and transformation. In 2017, the inorganic N from major rivers entering the sea reached 2.3×10^9 t in China, and the nitrate accounted for 91.3% (Bulletin of China Marine Ecological Environment Status 2017, 2018). Therefore, it is important to explore the source and transport of nitrogen in rivers to gain a deeper understanding of the nitrogen cycle and pollution prevention.

The Yellow River (YR) Basin, as an important core area for grain production in China, produced nearly 243 million tons of grain in 2022, accounting for 35.33% of the country's total grain production (Zhang et al., 2024). The YR Basin has wide prospects for agricultural development. Moreover, YR is the fifth largest river in the world, it has the second highest sediment load (Qs= 1.08 Gt·yr^{-1}) in the world (Milliman & Meade, 1983), with a suspended particulate matter (SPM) concentration 35 kg·m^{-3} (Wang et al., 2016). Through sampling and literature research, it was found that the lower reaches of the YR (LYR) presented significantly higher levels of both total dissolved N (TDN) and dissolved nitrate (D-NO_3^-) than the upper and middle reaches (Supplementary Fig. S1; Han et al., 2022). And the obvious difference between the LYR and other river sections is the annual water and sediment regulation (WSR) event and the agricultural activities in the washland soil around the LYR. On the one hand, due to the high SPM and low flow rate, a quarter of the SPM in LYR is silted each year (Yu, 2002). Therefore, to regulate the SPM and avoid flooding, WSR event has been implemented in LYR since 2002. Importantly, during WSR (2-3 weeks), roughly half of the entire annual sediment and water, as well as substantial amounts of terrestrial materials including N and phosphorus (P) are discharged into the YR Delta and Bohai Sea (Kong et al., 2020). Meanwhile, these substances are inevitably suspended, deposited, retransformed and distributed in the lower reaches of the YR during WSR (Xia et al., 2016). Previous study showed that the total N (TN) content of the channel sediments decreased by ~40% after WSR event in 2018 (Hou et al., 2021). However, the contribution of SPM and SS changes in the LYR triggered during WSR to the N concentration in LYR water column is currently unknown. Meanwhile, different N fractions in SPM and SS have different environmental significance and release risks (Wang et al., 2008).

On the other hand, the N in water column would be adsorbed by SPM and/or SS. It was shown that the adsorption of ammonia in the water column by sediments decreased with increasing depth (He et al., 2015), and the ammonia is more likely to be adsorbed by clay (Stevenson, 1986). However, nitrate is the dominant form of TN and has been recognized as the main pollutant in the YR, accounting for 95% of the TN. For the SPM in YR, it can influence the rates of nitrification and denitrification in the water column by providing attachment sites for bacteria (Xia et al., 2009). For SPM in YR, it can influence the rates of nitrification and denitrification in the water column and thus the nitrogen concentration in the Yellow River water column by providing attachment sites for bacteria (Xia et al., 2009). In addition, the soils around the YR, especially within the YR dam, will often be subject to agricultural activities, making it a unique "dam-soil-river" system. Therefore, agriculture around the LYR is highly dependent on irrigation from the LYR, and irrigation inevitably brings a large amount of SPM into the farmland to become top soil. Meanwhile, a portion of the irrigation water will carry N fertilizer back into the YR (Lv et al., 2024; Zhang et al., 2024). Therefore, it is of great significance to explore the contribution of the surrounding soils in the LYR to the transient increase in N concentration in the LYR. Meanwhile, the adsorption characteristics of SPM, SS and washland soil for nitrate, and the relationship between SPM, SS and washland soil and N pollution budget in the YR have rarely been studied. This is essential for understanding the ability of rivers with high SPM to transport N and the geochemical N cycling.

Therefore, in order to investigate the reasons for the abrupt increase of N in the LYR, N fractionation extraction, N adsorption, and N leaching methods of SPM, SS and soil have been used in combination. The purpose of this study was to (1) explore the effects of WSR event on the composition and distribution of N forms in water, SPM and SS in LYR; (2) clarify the adsorption characteristics of SPM, SS and soil on nitrate in LYR; (3) identify the effect of the soil N leaching on the increase of N in the LYR.

2. Materials and methods

2.1. Study area

The LYR is the mainstream sections below Taohuayu in Henan province to estuary in Shandong province, with a length of 786 km. The LYR account for only 3% of the total basin area, which is 23,000 km^2. Due to the high SPM concentration and historic low flow velocity in the YR, the LYR has been silted up for a long time to form the world-famous "Hanging River on the Ground". There are no larger tributaries in this section except for the Dawen River, which is fed by the Dongping Lake. Consequently, the alluvial plains of the YR created the soil surrounding its lower reaches, and the primary crops planted there were soybeans, wheat, corn, and cotton (Jiang et al., 2023).

These crops were grown on a biannual basis. Meanwhile, agricultural output, N fertilizer use and pesticide use in Henan and Shandong provinces, which are located in the LYR, are much higher than in other provinces through which the YR flows (Supplementary Table S1, Fig. S2 and Table S2). Furthermore, from June to July, Xiaolangdi Reservoir works with the Wanjiazhai and Sanmenxia Reservoirs to conduct an annual WSR program to clear up sedimentation in LYR. Statistics showed that since 2002, after 22 years of WSR, the YR has accumulated 3.25×10^9 t sand into the sea (Yellow River Sediment Bulletin 2022, 2023). The main channel of the downstream has been dropped by an average of 3.1 m. The WSR of the YR in 2023 was conducted from June 21st to July 11th, totally 20 days.

2.2. Sampling

Samples were collected before, during and after WSR because June-July is the rainy season of the YR and is the time of WSR and covers the peak period of agricultural activities such as planting, growing, fertilizing and irrigating. As shown in Fig. 1, 16 representative water and SPM samples (numbered S-1 to S-16, including 2 sampling sites of Dongping Lake) were collected at ~50km intervals. The sampling time was June 21-24, 2023 (before WSR), July 3-6, 2023 (during WSR) and July 20-22, 2023 (after WSR). For SS samples, only S-3, S-4 and S-9 SS sites before WSR, S-1 and S-4 during WSR and S-4, S-6, S-9 and S-16 after WSR were sampled due to current velocity and sampler limitations. Soil samples were collected from agricultural fields or washes where leachate could be discharged to the YR, depending on the topography of the field. They are S-2(1), S-2(2), S-2(3), S-5, S-6, S-9, S-12, and S-13. Specific sampling details are provided in Supplementary Material Text S1.

2.3. Sample analysis

The position, coordinates, temperature, dissolved oxygen, electrical conductivity, total dissolved solids, salinity, pH, oxidation reduction potential and the velocity of the river water current at sampling sites were analyzed in the field. Details about the basic water quality parameters and specific locations of sampling sites were presented in the

Supplementary information (Supplementary Table S3, S4 and S5). Sampling site position, coordinates and planting crop of soil samples were shown in Supplementary Table S6. The concentrations of SPM, TN, total nitrate-nitrogen (T-NO_3^-), total nitrite-nitrogen (T-NO_2^-), total ammonia nitrogen (T-NH_4^+), total reactive phosphorus (T-PO_4^{3-}), total P (TP), TDN, dissolved nitrate nitrogen (D-NO_3^-), dissolved nitrite nitrogen (D-NO_2^-), dissolved ammonia nitrogen (D-NH_4^+), dissolved reactive phosphorus (D-PO_4^{3-}) and total dissolved P (TDP) were measured in water samples. The pH, particle size, TN, total carbon (TC) and total organic carbon (TOC), and organic matter (OM) in SPM, SS and soil samples were determined. Specific details of the measurement are provided in Supplementary Material Text S1. In order to uniformly assess the different forms of N in SPM, SS and soils, the same methodology was used to extract their N fractions based on the method of Wang et al. (2009). TN in SPM, SS and soil is composed of transformable N (TFN) and non-transformable N (NTFN). TFN includes ion exchangeable form N (IEF-N), weak acid extractable form N (WAEF-N), strong alkali extractable form N (SAEF-N) and strong oxidant extractable form N (SOEF-N). The specific extraction agents and N fractions extracted were shown in Fig. S3 in the Supplementary information.

2.4. Adsorption experiments

Nitrate was chosen for the adsorption experiments because it accounts for more than 95% of the TN in the YR water column. Weighted 0.0300 g of sediment (SPM, SS and soil) and 30 mL of deionized water with a background electrolyte concentration of 0.05 $g \cdot L^{-1}$ NaCl were added to the seven polypropylene centrifuge tubes (SPM = 1 $kg \cdot m^{-3}$). A certain nitrate concentration solution was added to all centrifuge tubes except the blank centrifuge tubes (0-39.1 $mg \cdot L^{-1}$). The centrifuge tubes were placed in a shaker at 25°C and 250 rpm for 50 h. During the incubation period, the pH was maintained at 7.45 ± 0.05 (the average pH of the water samples is from 3 times of sampling) with 0.001 $mol \cdot L^{-1}$ NaOH. After 50 h, the tubes were centrifuged at 4000 rpm for 10 min, and the supernatant was filtered through a 0.45 μm filter to determine the nitrate concentration. Adsorption isotherms were plotted for 14 sites in all SPMs before, during and after WSR, 8 sites in SS and 6 sites in soils. The adsorption isotherms of S-1, S-3, S-6, S-12 and S-16 for SPM, S-4 (before WSR), S-7 (during WSR), S-9 (after WSR) and S-16 (after WSR) for SS and S-9 and S-13 for soils were selected to be shown as representatives in the text, and the rest were shown in Fig. S4 of the Supplementary information.

2.5. Leaching experiment

A range of SPM concentrations (1, 5, 10, 30, 50 kg·m^{-3}) were used to determine the concentration of nitrate released from the sediment to the overlying water column in leaching experiment. These SPM conditions were selected to provide a comprehensive analysis of known SPM changes in the YR over the past decades (Wang et al., 2016). The initial nitrate concentration range (0- 9.8 mg·L^{-1}) was provided by spiking with KNO_3 to simulate typical conditions of N input to the YR. The background electrolyte, pH, and shaking conditions of the experiment were the same as those of the adsorption experiment. The centrifuge tubes were centrifuged after shaking and then the supernatant was filtered through a 0.45 μm filter membrane and the nitrate concentration of the supernatant was determined. The S-9 site of SPM in before and after WSR, S-9 site of SS and S-9 and S-13 sites of soil were selected for experiments.

The leaching amount and leaching rate of nitrate from SPM, SS and soil were calculated as follows (Wiggenhauser et al., 2024):

$$A = (C \times V)/M \tag{1}$$

$$R = A/D \tag{2}$$

Where: A is the amount of leaching, mg·g^{-1}; C is the nitrate concentration measured in the leachate, mg·L^{-1}; and V is the volume of the leachate, L; M is the mass of SPM, kg; R is the leaching rate, %; D is the TN content of SPM, mg·g^{-1}.

3. Results

3.1. Changes of SPM in LYR caused by WSR event

The concentration of SPM before, during and after WSR event was shown in Fig.

2(a). The concentration of SPM in the LYR increased slightly in WSR period (the average SPM before, during and after WSR were 1.66, 1.68 and 1.32 kg·m^{-3}, respectively). As shown in Fig. 2(b), the particle size of soil is obviously larger than that of SPM and SS. The proportions of clay, silt and sand with SPM before WSR were 5.87%-8.54%, 74.67%-85.27% and 6.81-17.23%, respectively. The SPM in before and during WSR mainly composed of silt, whereas the SPM after WSR consists mainly of clayey silt. For SS, it contained significantly more coarse-grained sediments (silt 61.34% + sand 33.33%) than SPM. In addition, the particle size of the soils had a clear geographical character. Sand (average 54.34%) dominated the soils at site S-2, while silt (average 70.20%) dominated the rest of the sites.

3.2. N changes in LYR in WSR period

3.2.1. Distributions of N and P in LYR water column in WSR period

As shown in Fig. 3, compared with before WSR, the concentrations of dissolved N after WSR showed a downward trend. The order of magnitude of the average concentration of D-NO_3^-, D-NH_4^+, D-NO_2^-, and TDN was (unit: mg·L^{-1}): before WSR (3.41) > during WSR (2.76) > after WSR (2.41), during WSR (0.041) > after WSR (0.023) > before WSR (0.020), during WSR (0.00113) > after WSR (0.00107) > before WSR (0.00015) and before WSR (4.07) > during WSR (3.40) > after WSR (3.09), respectively (Fig. 3(a)-(d)). Overall, the N concentration in the LYR decreased after WSR. The trends of total form N and dissolved N were consistent, both of which showed that they were higher before and during WSR than after WSR (Fig. S5). The following were the sequence in which the average TDP and TP concentrations declined (unit: mg·L^{-1}): during WSR (0.0131) > after WSR (0.0124) > before WSR (0.0076) and during WSR (1.42) > before WSR (1.38) > after WSR (1.08), respectively (Fig. 3(e), (f) and Fig. S5). These trends indicated that the P concentration in the particulate state was obviously higher than that in the dissolved state.

3.2.2. Changes of N fractions content of SPM, SS and soil in the LYR

Fig. 4(a) showed the distribution of the five N fractions in SPM, SS and soil under the influence of WSR event. IEF-N, WAEF-N, SAEF-N and SOEF-N are all transformable N (TF-N). In brief, TF-N is the portion of sedimentary N that can actually participate in the N cycle, and be released and reengaged during marked changes in the depositional environment. The average content of different N fractions in SPM before, during and after WSR were arranged as follows (unit: $mg \cdot g^{-1}$): WAEF-N (0.116) > NTF-N (0.090) > SOEF-N (0.077) > IEF-N (0.021) > SAEF-N (0.017) for before, NTF-N (0.149) > WAEF-N (0.091) > SOEF-N (0.088) > SAEF-N (0.070) > IEF-N (0.011) for during and NTF-N (0.475) > SOEF-N (0.199) > WAEF-N (0.077) > SAEF-N (0.027) > IEF-N (0.012) for after WSR, respectively. In the SPM in LYR, the most readily released N, IEF-N, accounted for only 6.69% of the TN in SPM before WSR (during WSR: 2.71%; after WSR: 1.49%). For SS, similar to SPM, NTF-N, SOEF-N, and WAEF-N, which together accounted for more than 80% of TN content, were the predominant N fractions in SS. Compared with SPM and SS, the N content in the soils were relatively high, which may have been due to the agricultural pollution. The sites with TN content exceeding 0.550 $mg \cdot g^{-1}$ accounted for 56.25% of the total sites. This is notably true for the S-5 and S-13 sites, which have TN content of up to 1.122 $mg \cdot g^{-1}$ and 1.547 $mg \cdot g^{-1}$, respectively. The average contents of several N fractions in the soils over the WSR period were organized as follows (unit: $mg \cdot g^{-1}$): NTF-N (0.445) > SOEF-N (0.194) > IEF-N (0.092) > SAEF-N (0.026) > WAEF-N (0.006). The soil had a substantially larger content of IEF-N, which was 6.24 and 10.77 times higher than in SPM and SS, respectively. In particular, the IEF-N content of S-2 (2) (0.192 $mg \cdot g^{-1}$) and S-13 (0.371 $mg \cdot g^{-1}$), which accounted for 21.28% and 23.95% of the TN, was 12.98 and 25.10 times higher than the IEF-N of SPM. In addition, TC, TN, TOC, and OM exhibited essentially the same trend in SPM, SS and soil in the YRL (Fig. 4(b)).

3.3. Multivariate analysis between SPM and N in water column, SPM, SS and soil

The redundancy analysis and Pearson correlation of water, SPM, SS and soil to environmental factors were shown in Fig. 5 and Fig. S6. In the LYR water column, dissolved N was strong positively correlated with salinity and essentially independent of SPM, whereas SPM and silt had a positive effect on TN and TP. This was consistent with the results of the correlation analysis, indicating that a higher SPM concentration and proportion of silt can accumulate more particulate N and P rather than dissolved. Moreover, each form of N was positively influenced by clay, silt, OM, TOC and TC in SPM, SS and soil. It showed that the smaller the particle size in SPM, SS and soils in the LYR, the more N in different forms is carried, while OM and carbon have significant effects on the distribution of different forms of N.

3.4. Adsorption isotherms of nitrate in SPM, SS and soil

The adsorption isotherms of nitrate in SPM, SS and soil were shown in Fig. 6. It can be seen from Fig. 6(a)-(d) and S3 that the correlation coefficient (R^2) obtained by the adsorption curves of SPM and SS for nitrate was low (<0.09). It resulted in the inability to determine ENC_0 and adsorption equilibrium constants (K) for the nitrate adsorption curves for SPM and SS. Compared to SPM and SS, the soil presented a good correlation between the adsorption equilibrium concentration (C_{eq}) and adsorption amount of nitrate (Q) when it was used as a SPM ($R^2 > 0.94$) (Fig. 6). The K, EPC_0 and standard δ-values for all four soil samples from different depths of S-13 and S-9 were presented in Table 1. Soil S-13 were all sources of nitrate in the water column, while S-9 were all sinks for nitrate in the water column.

3.5. N leaching characteristics of SPM, SS and soil

To explore the abrupt increase in N concentration in LYR, leaching studies on SPM, SS, and soils were carried out. Site S-9 is located at the Henan-Shandong province junction and downstream of the junction of Dongping Lake and the YR. At site S-13, both IEF-N and TN in the soil were the highest, while both IEF-N and TN were at lower levels at site S-9. To conduct the research, site S-9 for SPM and SS, as well as S-9 and S-13 for soil were selected. Laboratory simulations showed that nitrate concentration in the water column of the LYR was not significantly affected by the reduction of SPM and SS in the SPM range of 0-50 $kg \cdot m^{-3}$ (Fig. 7 (a-d)). In addition, as shown in Fig. 7 (e), it was crucial to mention that soil S-9 was identical to SPM and SS when the soil surrounding the LYR was utilized as SPM in the experiment. It also had essentially little impact on the nitrate concentration in the water column. However, at soil S-13, the leaching nitrate increased with the increase of SPM (Fig. 7 (f)). In addition, for soil S-13, the initial nitrate concentration (exceeded 3.7 $mg \cdot L^{-1}$) restricted the nitrate leaching rate (R) when the SPM concentration was 5 $kg \cdot m^{-3}$ or less, as the Fig. 8 illustrated. It means that high nitrate concentrations in the water column inhibit the migration of nitrate from soil into the water column. However, in the actual YR water column, the concentration of D-NO_3^- basically remained ~2.5 $mg \cdot L^{-1}$, which would facilitate the migration of N from the soil to the water column. Furthermore, when the SPM concentration was 10-30 $kg \cdot m^{-3}$, the leaching rate of nitrate was in the range of 14.97-16.18%, which did not vary obviously with the SPM and initial nitrate concentrations. It suggests that changes in SPM levels do not inhibit the release of nitrate from the soil to the water column.

4. Discussion

4.1. N remaining from agricultural activities in washland that would be eluted away by WSR event

Compared to before WSR, the SPM in LYR kept narrow ranges during and after WSR respectively, indicating that the effect of WSR events on the SPM in the YR was not remarkable. Meanwhile, there was no correlation between SPM and D-NO_3^- and TDN (Fig. 5), indicating that the elevated N concentration in the LYR was not caused by the variation of SPM concentration. The WSR period in 2023 was from June 21 to July 6, 2023, while the sampling period of this experiment was from July 2 to July 4, 2023 during WSR. Thus, water samples were not collected during the sand transfer period, meaning that water samples with high concentrations of SPM were not collected. This was demonstrated by the ongoing monitoring of SPM concentrations at the Xiaolangdi station from June 17 to July 10 and at the Lijin station from June 19 to July 16 in 2014 (Li et al., 2017). Moreover, SPM concentrations in LYR in WSR period were all less than 3 $kg \cdot m^{-3}$. It is well below the SPM concentrations before WSR in 2014 (4-

6 kg·m^{-3}, Li et al., 2017) and the SPM concentrations in 2020-2021 (7.19 kg·m^{-3}) (Liu et al., 2024). This change was consistent with the inference, but the YR still had a higher SPM than other rivers (Wang et al., 2016).

Overall, dissolved inorganic N (DIN) in YR was primarily composed of D-NO_3^-, which accounts for >90% of DIN. This was consistent with the results in the Ganges-Brahmaputra-Meghna River system in the non-industrial active area (Jiang et al., 2023). It suggested that N pollution in the YR mainly originated from urban sewage and fertilizer pollution. In addition, the Xiaolangdi Reservoir can have an artificial-lake effect on the river, resulting in anaerobic bottom waters in the reservoir. The previous study revealed that water in Xiaolangdi Reservoir showed an increase in NH_4^+ and NO_2^- and a decrease in NO_3^- compared to the YR water (Li et al., 2017). In this work, the sampling time before WSR was from June 21 to 24, 2023, when Wanjiazhai, Sanmenxia and other reservoirs had begun to prepare for WSR event. Therefore, the water flow of LYR increased and the water level rose before WSR. Meanwhile, the surrounding area is flooded by the river water, including a portion of the farmland that is inside its path (Bi et al., 2014; Tao et al., 2010). Studies have shown that floodplain inundation events on floodplains can dissolve almost all nitrate residuals from agricultural activities into the water column, causing D-NO_3^- and TDN to rise in LYR before and during WSR (Salazar et al., 2014). This is the main reason for the positive correlation between salinity and D-NO_3^- and TDN in the water column (Fig. 5(a)). Therefore, riverine agricultural activities in the LYR do increase D-NO_3^- and TDN concentrations in water column, which was consistent with previous findings (Hou et al., 2021; Li et al., 2017). Unlike N, D-PO_4^{3-} decreased slightly before and during WSR compared to after WSR. It was mainly attributed to the fact that in the Xiaolangdi Reservoir, D-PO_4^{3-} was adsorbed or enclosed on SPM, deposited in the reservoir, and removed from the water column during non-WSR periods (Wang et al., 2007). D-PO_4^{3-} decreased during water transfers when large amounts of low D-PO_4^{3-} water were released from Xiaolangdi Reservoir in WSR.

4.2. Far greater potential in soil to release reactive N to the LYR than SPM and SS

In this work, the N fractions in SPM, SS and soil was analyzed using an the promoted Ruttenburg's sequential extraction process in sediments (Ruttenburg, 1992; Wang et al., 2008). The method extracts the N into five fractions: IEF-N, WAEF-N, SAEF-N, SAEF-N and NTF-N. Overall, the TN content of soils around the LYR basin was higher than the TN content of SS and SPM in LYR, except for the SPM after WSR. Similarly, soil TN content was significantly higher ($p < 0.001$) than sediment in the Yangtze River Basin ($p < 0.001$) (Yang et al., 2023). It demonstrated the tremendous potential for N in soils around large rivers to be released into river waters. Since one of the purposes of WSR in the YR was to flush the channel sediments of LYR, the channel sediments are the main component of SPM in the LYR in before and during WSR. It implied that a natural river screening process has been applied to SPM before and during WSR. Meanwhile, SS were mainly derived from SPM that was screened and deposited in the channel at different stages before WSR. Consequently, SPM in before and during WSR and SS have higher particle sizes than after WSR (Fig. 2(a) and (b)). The soil around the LYR mainly consisted of alluvial deposits from the YR (Jiang et al., 2023). In addition, another portion of the top soil around the LYR came from SPM from the LYR due to the effects of irrigation (Fig. S7). Meanwhile, pollution from agriculture and industry would result in high levels of TF-N and NTF-N in the soils than in the SPM from the LYR.

Generally, IEF-N is a weakly adsorbed N, it is highly connected with environmental parameters that influence N adsorption in sediments (Wang et al., 2008). In this work, IEF-N was found the most readily released N portion from sediments. Except for SPM at S-12 before WSR ($0.072\ mg\cdot g^{-1}$), IEF-N contents were extremely low and steady in SPM and SS, all below $0.033\ mg\cdot g^{-1}$. This was much lower than the IEF-N content in soils around the YR (0.002-$0.464\ mg\cdot g^{-1}$ with an average of $0.092\ mg\cdot g^{-1}$). However, compared to the IEF-N in shallow lakes in the middle and lower reaches of the Yangtze River ($0.096 - 0.196\ mg\cdot g^{-1}$), the YR presented higher reduced

IEF-N (Wang et al., 2008). Because WAEF-N is linked to carbonates and OM, so the content of WAEF-N in sediments mainly depends on the carbonate content and the change of pH during the mineralization of OM. Its distribution was similarly characterized by high OM content in SPM and SS in the YR and low OM content in soil (Fig. 4(b)). The WAEF-N in the soil was lower than that in the SPM and SS, which contradicted the distribution of IEF-N. Furthermore, the soil presented high pH (7.54-8.37) (Table S7), which was detrimental to the preservation of OM, resulting in low WAEF-N. Furthermore, the SAEF-N and SOEF-N in SPM after WSR were close to those in soil. Generally, the SAEF-N represents N bound to iron and manganese oxides, the amount of which is largely controlled by the redox environment of the sediment (Kozerski Kleeberg, 1998). Research has demonstrated that inorganic N, particularly NH_4^+-N, can be adsorbed by iron oxide surfaces (Balzer, 1984). However, in this work, NH_4^+-N dominated the SAEF-N of SPM after WSR with 59.93%, while in soil NO_3^--N was the main component of SAEF-N with 62.31%. It suggested that the soil was more oxidizing compared to SPM after WSR. This was consistent with changes in OM and TOC content in soil and SPM (Fig. 4(b)). Because the SOEF-N is a form of N that is bound to OM and sulfides and is thought to be composed of organic N (Lv et al., 2005), it aways closely relates to fine-size particulate matter (Zhao et al., 2020). Therefore, fine sediment particles would be helpful to create an impermeable anaerobic environment that favored the preservation of OM. This corresponded to elevated OM in SPM after WSR (107.40 $mg \cdot g^{-1}$). However, the OM content in the soil kept stayed modest (39.54 $mg \cdot g^{-1}$). Therefore, the input of exogenous organic N is the main reason for the low OM and high organic N in the soil around the LYR. This finding revealed that the potential for reactive N release from soils around the LYR was much greater than that from river SPM and SS.

4.3. Comparison of DIN/ dissolved inorganic P (DIP) ratio and DIN concentration with other major world rivers

Large rivers are the main interface between the terrestrial and marine environments and play a crucial role in the global N cycle (Dagg et al., 2004). The YR is 5,464 km long, originating on the Tibetan Plateau and flowing through the Loess Plateau, the North China Plain, and finally into the Bohai Sea. Consequently, the YR carries a large amount of SPM and land-based nutrients into the Bohai Sea each year. As shown in Table 2, it was found that the DIN concentration in the YR was obviously higher than those in Yangtze River, Mississippi and Rhoˆne River. This is linked to human activities in the watershed, including the use of more cropland, the application of fertilizer to agriculture, and the potential discharge of sewage (Liu et al., 2012). Nevertheless, the DIN/DIP in the YR is the highest compared to other rivers, especially before WSR, the DIN/DIP value reach to 6902. Even after WSR, this ratio is still more than 10 times higher than that in the Yangtze River and 22.8 and 280.2 times higher than that in Rhoˆne River and Yenisei River respectively. It means that the YR is an extremely P-limited ecosystem in its lower reaches. This phenomenon can be explained by the high-intensity irrigation and soil erosion in LYR have brought more N fertilizer into the river (Wu et al., 2021). The high population density in Henan and Shandong provinces (577 and 638 people·km^{-2}) may be the primary cause of the greater DIN concentration in the YR (2.493 mg·L^{-1}) compared to the Yangtze River (1.750 mg·L^{-1}) (Zhang et al., 2024). Previous studies have shown that N concentrations in rivers are strongly correlated with regional population density (Chen et al., 2003). Another reason is the SPM concentration in the YR is as high as 35 kg·m^{-3}, which is the highest in the world (Wang et al., 2016). Meanwhile, P is easily to be adsorbed or trapped in SPM due to its high concentration and the presence of reactive iron in the YR (Pan et al., 2013). It has led to extremely low DIP concentrations (0.0025 mg·L^{-1} after WSR) in the water column. Thus, high N and low P make the YR a unique river with the highest DIN/DIP ratio in the world.

4.4. Role of SPM, SS and soil as a source or sink for N in the LYR

Previous studies reported that equilibrium ammonium concentration could be used to investigate the adsorption and release characteristics of ammonia in SPM and sediments (He et al., 2015). In this work, equilibrium nitrate concentration (ENC_0) was attempted to explore the contribution of SPM, SS and soil to nitrate in LYR water column. The adsorption curves of SPM and SS for nitrate could not be determined ENC_0 was associated with fewer active nitrate absorbable sites in SPM and SS. According to the presence of nitrate in SPM and SS, nitrate mainly exists in SOEF-N while least exists in IEF-N. In general, the order in which each N fraction is released correlates with how strongly it is bonded to the sediment. The looser the bond, the easier it is to release. Therefore, the order of release of different N fractions was IEF-N > WAEF-N > SAEF-N > SOEF-N. The adsorption isotherms of S-7 and S-8 in SS, however, were still challenging to fit, despite not being low in IEF-N (S7: 0.021 $mg \cdot g^{-1}$; S-8: 0.033 $mg \cdot g^{-1}$). This phenomenon suggested that it was basically difficult to rely on increasing or decreasing the amount of SPM to remove nitrate from water column in short time. However, it has been shown that after one month of closed incubation, SPM at a concentration of 1 $kg \cdot m^{-3}$ can increase N loss from YR flows by 25-120%, which rises with the high TOC content (Xia et al., 2017). This is owing to the fact that SPM, when in contact with aerobic fluids, forms internal anoxic zones that increase nitrate denitrification and N_2 production, resulting in riverine N losses. Therefore, it is challenging to remove nitrate from the YR water column only by SPM in the short term.

For soil, the planted crop (corn) was already at the staminate stage when S-9 was sampled on July 23, 2023, and fertilization activities would not typically be performed at that time. Therefore, the content of active N in soil S-9 at this site was relatively low (Fig. 4(a)), which became the sink of nitrate in the YR. However, S-13 was sampled on June 23, 2023, when the crop (corn) was in the pulling stage, shortly after fertilizer application and high in reactive N. Similarly, S-2(2) and S-6 have relatively high reactive N content. However, some of the soil will only be washed into the LYR during WSR period, when the level of water in the river rises drastically. During non-WSR periods, SPM and SS are the primary components of particulate matter in the YR, but they can hardly remove nitrate by adsorption resolution.

4.5. Effects of SPM, SS and soil N leaching on the N cycle in the YR

In the early 1960s, DIN concentrations in the YR were generally below 1 mg·L^{-1} (Yu et al., 2010). By the 1980s, DIN concentrations had risen dramatically. During the period of 1980-2012, the DIN in YR ranged from 1.61 to 4.98 mg·L^{-1}, which was relatively high (Wang et al., 2018). Up until 2000, there was a steady increasing tendency, but after that, it was kept at a comparatively high level. Following 2012, the Chinese economic growth strategy pushed the idea of green development, and witnessed an enormous decrease in the amount of DIN in the YR (Xiao & Zhao, 2017). Currently, the DIN concentration in the YR is maintained at about 2.5 mg·L^{-1}.

At S-9, reductions in SPM, SS, and soil had a negligible effect on nitrate concentrations in the LYR water column. This could be attributed to the poor the surface reactivity of the solids to nitrate (Pan et al., 2013). Furthermore, SPM and SS exhibited the same leaching pattern before and after WSR. It implied that the influence of WSR event on nitrate adsorption by SPM was restricted. It was discovered that a decrease in SPM contributes to a rise in P and ammonia in the water column by comparing the adsorption isotherms of these three elements by SPM (Pan et al., 2013; He et al., 2015). Particularly on clays, ammonium and P have the tendency to adsorb onto inorganic particulate matter (Stevenson, 1986). However, soil S-13 is entirely different from S-9. Based on the composition of different N fractions in the soil S-13, WAEF-N content was the lowest (0.010 mg·g^{-1}), and SAEF-N and SOEF-N content were comparable to SPM after WSR (Fig. 4(a)). Soil S-13 differed from the N forms of SPM and SS mainly by IEF-N and NTF-N, with NTF-N being difficult to engage in the biological N cycle. Therefore, the N leached from soil S-13 was mainly IEF-N. The leaching rate of nitrate in soil S-13 was estimated to be 14.74% on average, whereas leaching of IEF-N accounted for 19.25% of TN and 76.56% of leached IEF-N. Taking the YR dam as the boundary, a total of 1160.35 (one side) × 2 km^2 of cultivated land along the YR dam to the YR from the beginning sampling site to the YR estuary was assessed (Fig. S8). The thickness of the top soil (OM layer 0-10 cm and humus layer 10-25 cm) was 25 cm.

Studies have shown that the mean value of soil density in the North China Plain is 1.425 $g \cdot cm^{-3}$ (He et al., 2010). By calculation, the surface soil around the YR has 5.787×10^4 t of N that is extremely easy to release to the environment. Taking the runoff volume at Lijin station in 2022 as a representative (260.90×10^8 m^3) (Yellow River Water Resources Bulletin 2022, 2023), releasing all easily released N from the top soils around the YR can increase the N concentration in water column by 2.218 $mg \cdot L^{-1}$. It has been demonstrated that an increase in the use of synthetic N fertilizers is strongly correlated with an increase in nitrate in surface waters (Howarth et al., 1996; Donoso et al., 1999). A long-term tracer study revealed that 30 years after isotope-labeled N fertilizer was put to agricultural soils in 1982, 8-12% of the fertilizer N was leaching into the hydrosphere. Furthermore, it is expected that part of the N fertilizer left in the soil will be absorbed by crops and leak into groundwater as nitrates for at least another 50 years (Sebilo et al., 2013). It implied that N would be continuously released into the hydrosphere for eight decades following a single application of N fertilizer to unfertilized N soil. However, in this work, soil fertilizer applications have continued to increase since the 1980s as irrigated areas and the pesticide industry have grown tremendously (Fig. S2). While N fertilizer applications slowly declined after 2015, compound fertilizer applications steadily increased (China Statistical Yearbook 2023, 2023). Meanwhile, Henan and Shandong, located in LYR, are major agricultural provinces in China. Furthermore, the applications of N and compound fertilizers of Henan and Shandong are much higher than that of other provinces around the YR (China Statistical Yearbook 2023, 2023). It means that the N and compound fertilizers that have been applied to farmland since the 1980s are constantly and continuously released into surface water. In addition, the contribution of N-containing pesticide applications to soil N levels is quite considerable. The soil around the YR shows clear signs of pesticide application (Fig. S9). According to a survey by the National Bureau of Statistics of China, pesticide use in Shandong (120,342 t) and Henan (107,231 t) Province was higher among all provinces in the country (Table S2). And, most of the

N-containing pesticides are carbamate pesticides, which decompose easily in alkaline soils and can easily enter the hydric environment (Heinzen & Cesio, 2024). Meanwhile, the nitrification rate in the YR was aided by the rise in SPM in the lower sections of the river, resulting in NO_3^- being the predominant form of N in the water body (Hu et al., 2023; Xia et al., 2009). In addition, the total precipitation per unit area in Henan (2182.95×10^{-5} m·km^{-2}) and Shandong (6296.08×10^{-5} m·km^{-2}) provinces in LYR is much higher than that in the other provinces (except Sichuan province) through which the YR flows (Table S8). It provides a very suitable base condition for nitrate leaching from soils in the LYR. Surface runoff coefficients for flat cropland are 0.45-0.60. It was reported that, during the rainfall period, nitrate leaching from soils cropped with maize, spring barley and faba bean was 1-31 (during maize growth), 67 and 63 kg·ha^{-1}, respectively. Therefore, the continuous leaching of N from the soils around the YR will considerably increase the N concentration in LYR. So, the key step to reduce N load in LYR is to control soil agricultural pollution.

4.6. Limitations

N fractionation extraction, N adsorption and leaching analyses of SPM, SS and soils in the LYR clarified that agricultural activities in washland soils do cause a transient increase in N concentration in the LYR. However, there are still several challenges need to be addressed to fully understand the N cycle in the LYR. Firstly, although it has been qualitatively determined that N leaching from washland soils increases N concentrations in the LYR, quantitative analysis is needed. Secondly, ammonia, nitrite and organic nitrogen are also important components of N in terrestrial waters, their contribution need to be identified. Thirdly, the role of microorganisms in the nitrogen cycle in the LYR should been clarified in future work.

5. Conclusion

The YR is an important carrier of N terrestrial-sea transport and transformation. This study showed that the N concentration in LYR before and during WSR was clearly elevated, compared to that after WSR. This was primarily induced by agricultural activity on the river channel. In addition, more N is readily released from soil to the environment. Adsorption experiments showed that for the current SPM concentration (~ 1 kg·m^3), the adsorption and resolution of SPM could not lead to a sudden increase in N concentration in the LYR. Soil flushed into the YR due to water and sand transfer will have a little regulating effect on N concentration in the LYR. The high activity N soil site (S-13) showed a source of nitrate from the YR, and the low activity N soil site (S-9) showed a sink of nitrate from the YR. The variation of SPM had no remarkable effect on nitrate concentration in the water column due to the poor surface reactivity of solids to nitrate. While the estimated leaching rate of nitrate in soil S-13 averaged 14.74%, IEF-N accounted for 19.25% of TN, of which 76.56% was leached. Therefore, the continuous leaching of N from soils around the LYR would be the main for the notably increase the N concentration in LYR. So, more attention should be paid to agricultural N pollution in the management of N concentration in LYR.

Acknowledgements

This study was supported by the Key Project of Shandong Provincial Natural Science Foundation (Grant No. ZR2020KE048), and the Strategic Priority Research Program of the Chinese Academy of Sciences (Grant No. XDA23050203).

CRediT authorship contribution statement

Na-na Hu: Writing – original draft, Visualization, Software, Methodology, Formal analysis, Data curation, Conceptualization. **Yan-qing Sheng:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Funding acquisition. **Zhao-ran Li, Zheng Wang, Wei-han Xu** and **Huiyi Yang:** Investigation, Data curation.

Data Availability Statement

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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