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Ammonium adsorption on particles with different grain sizes sieved by sediment resuspension

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ABSTRACT

Sediments can be sieved into groups of particle size during hydrodynamical resuspension, potentially impacting the nutrient biogeochemical processes. This research addressed the ammonium adsorption on particles with different grain sizes sieved by sediment resuspension. Five samples of surface sediments were collected from the Beijing–Hangzhou Grand Canal in the upstream areas of the Taihu basin, which is characterized by frequent sediment resuspension and heavy nitrogen (N) pollution. The N pollution of the sediments gradually increased from the upstream to downstream samples with increasing urbanization. Sediments at one of the five sampling sites were suspended and sieved into five grain size groups (50–150, 30–50, 10–30, 50–10 and <5 μm), which were used to conduct isothermal adsorption and sorption kinetics of ammonium (NH_4-N). The NH_4-N adsorption abilities of the particles become stronger as the particle size decreases. The adsorption of NH_4-N on particles <10 μm is characterized by a higher adsorption capacity (Q_e) and a stronger adsorption affinity energy (k_e) and lower zero-adsorption equilibrium N concentrations (ENC_0) were compared for coarser particles (>10 μm). However, the NH_4-N adsorbed on the particles could be nitrified into nitrate (NO_3-N) under aeration due to frequent disturbance in canals.

Keywords: Sediment resuspension; Particle size distribution; Ammonium adsorption; Nitrogen pollution

1. Introduction

Sediment resuspension plays an important role in the nutrient exchange between particles and water, during which the nutrient biogeochemical cycle is active [1,2]. Nutrient exchange by sediment resuspension occurs widely in estuaries, canals and shallow lakes due to disturbances by hydrodynamics or zoobenthos [3–5]. This phenomenon is actually a sorting process of sediment particles with different grain sizes during sediment resuspension, transport and resedimentation. Fine particles, by resuspension, usually remain longer in the overlying water than coarse particles and tend to migrate longer distances downstream. Additionally, the particle sieving action may cause the redistribution of nutrients between particles of different sizes [6]. Therefore, clarification on the effect of particle sizes on nutrient exchange in the sediment–water interface could provide deep insight into the nutrient biogeochemical cycle and related environmental impacts.

The current studies on nutrient exchange at the sediment–water interface usually treat sediments as homogenized suspended particulate matter and are focused more on the sedimentary element of phosphorus than the soluble element of nitrogen (N). Ammonium (NH_4-N) is the main fraction of exchangeable N at the sediment–water interface [7,8]. It is mainly produced by the mineralization of sediment organic N or adsorbed from overlying water by sediments [9]. NH_4-N adsorption by suspended particulate matter is important for allowing the release or transformation of biological N.

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Therefore, we have two objectives in this research: (1) to characterize the N composition of group-sized sediment particles that experience frequent resuspension and (2) to investigate the impact of particle grain sizes on ammonium adsorption behaviours. This research could contribute to an accurate estimation of the ammonium nitrogen flux of rivers flowing into lakes and provide detailed information on the N transformation for particulate matter experiencing frequent resuspension.

2. Materials and methods

2.1. Water, sediment sampling and analysis

The research area is located upstream of Lake Taihu (Fig. 1(a)), the third largest freshwater lake in China, which is characterized by dense plain river networks in the Yangtze River Delta. More details on the local climate, topography and hydrology can be found in a related reference [6]. Five sampling sites (J1–J5) on the Zhenjiang-Wuxi Section of Beijing–Hangzhou Grand Canal were set to collect and analyse the samples of water and sediments (Fig. 1(b)). The Beijing–Hangzhou Grand Canal is characterized by the traffic of vessels between cities, with very frequent disturbance of the water and sediment layers.

The sampling trip was conducted in January of 2016 to investigate the water quality and sediment properties. Surface water samples were collected using a 5 L Plexiglass water sampler, and the water temperature, pH and dissolved oxygen (DO) values were measured on-site with YSI electrodes (Xylem Company, New York, USA). The water samples were immediately packed into refrigerated boxes and moved to the laboratory for storage and further analysis. The main water quality parameters that were analysed included total nitrogen (TN) and the inorganic fractions of nitrate (NO_3-N), nitrite (NO_2-N) and NH_4-N, permanganate index (COD_{Mn}) and total suspended solids (TSS). The samples for the soluble parameters were filtered through 0.45 μm cellulose acetate filters. The TN was determined by using a UV colorimetric method after alkaline persulfate digestion. NO_3-N and NH_4-N were analysed by ion chromatography (ICS-2000, Dionex Company, USA), and NO_2-N was determined using diazotization with sulfanilamide dihydrochloride [10].

Surface sediment samples were collected with a Peterson gravity sampler. All sediment samples were stored in air-sealed plastic bags and transported immediately to the laboratory. Sediment samples were air-dried at room temperature, homogenized and sieved through a stainless-steel sieve with a mesh size of 150 μm and then stored in the dark prior to further analysis [6]. The chemical properties analysed included oxidizable matter (OM), TN and the exchangeable fractions of NH_4-N, NO_3-N and NO_2-N. The TN concentration was determined by the Kjeldahl method, and OM was colorimetrically determined after treatment of the sample with chromic acid/H_2SO_4 according to the Walkley–Black method [11]. The exchangeable fractions of NH_4-N, NO_3-N and NO_2-N on the sediments were extracted with 2 mol·L^{-1} KCl [12] and determined with the same methods that were used for the water quality analysis.

2.2. Particle separation

Surface sediments Site J2, where the sediments experienced repetitive suspension and sedimentation due to strong hydrodynamic disturbance by ship propellers, was selected to analyse N fractionation and the adsorption of sediments as a function of particle size distribution. Five groups of different sized particles were obtained in the ranges of <5, 5−10, 10−30, 30−50 and 50−150 μm. The details of the particle separation are provided in a related reference [6]. The particle sizes of the sediments were analysed by a Mastersizer 2000 laser particle size analyser. All the obtained size-grouped sediment samples were air-dried and sieved through a stainless-steel sieve with a mesh size of 150 μm.

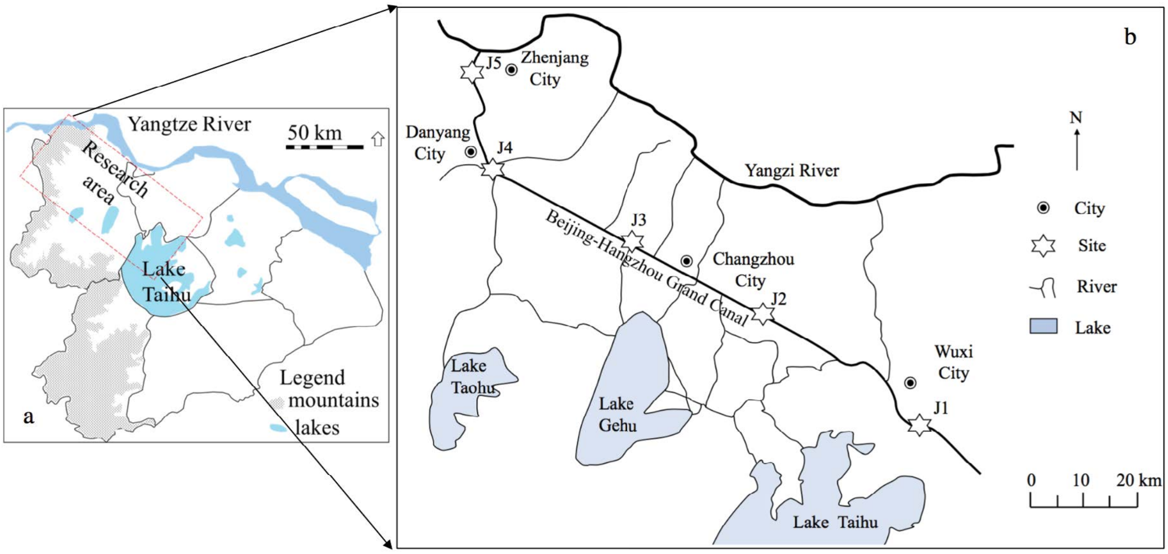

Fig. 1. (a) Locations of the sampling sites in the Beijing–Hangzhou Grand Canal, and (b) The upstream river networks the Taihu basin, China.

2.3. Isothermal adsorption of NH_4-N

Eight solutions, with NH_4-N concentrations of 0, 0.05, 0.1, 0.2, 0.4, 0.8, 1.6 and 2.0 mg L^{-1} were prepared for isothermal experiments. A mass of 0.25 g of sediment was mixed with 25 mL of N-spiked solution in 50 mL polypropylene centrifuge tubes and shaken at 25°C ± 1°C to reach equilibrium. The suspensions were then centrifuged, and the supernatants were passed through 0.45 μm cellulose acetate membrane filters [13]. The NH_4-N concentrations in the solutions were determined with the same methods that were used for water NH_4-N analysis of the water quality. The amount of adsorbed N was then calculated in terms of the difference between the initial amount of N added and the amount in the equilibrium solution. Each group of sorption experiments were performed in triplicate sets. The Henry linear fitting equation was used to fit the experimental data of the isothermal adsorption of the sediments on NH_4-N [14]:

$$Q_e = k_e \mathrm{ENC} - b \tag{1}$$

where Q_e is the amount of sorbed N at the equilibrium states (mg kg^{-1}), ENC is the equilibrium N concentration (mg L^{-1}), k_e is the N-binding energy constant reflecting the sorption affinity (L kg^{-1}) and b is the native adsorbed N (mg kg^{-1}). The intersection of the fitted linear equation with the X-axis is the equilibrium N concentration at the zero sorption.

2.4. Sorption kinetics of NH_4-N

The experiments for NH_4-N sorption kinetics were conducted by mixing 0.25 g of sediments with 25 mL of 2.0 mg L^{-1} NH_4-N solution in 50 mL polypropylene centrifuge tubes. Water samples were taken at different time intervals (5 min, 10 min, 30 min, 30 min, 1 h, 2 h, 3 h, 4 h, 6 h) and were filtered through 0.45 μm cellulose acetate membrane filters to determine the NH_4-N concentration [15]. The amount of adsorbed NH_4-N was determined in the same way as described for the isothermal experiments. Each group of sorption experiments were performed in triplicate sets. The adsorption kinetics fits a pseudo-first-order kinetic equation [16]:

$$Q_t = Q_e\left[1 - \exp\left(-k_1 t\right)\right] \tag{2}$$

where Q_e is the amount of sorbed N in the equilibrium states (mg kg^{-1}), k_1 is the first-order constant of sorption kinetics (h^{-1}) and Q_t is the amount of sorbed NH_4-N at time *t* (h).

The Pearson correlation coefficients, at confidence levels of 95% ($p < 0.05$) and 99% ($p < 0.01$), were used to analyse the correlation between the NH_4-N adsorption isotherms and the chemical properties.

3. Results and discussion

3.1. Water quality and sediment properties in the Beijing–Hangzhou Grand Canal

The water quality of the Beijing–Hangzhou Grand Canal was weakly alkaline with high DO concentrations. Under frequent shipping disturbances, the TSS concentrations were elevated, in the range from 69 to 121 mg L^{-1}. The N pollution is considerable in the river, with TN concentrations ranging from 3.3 to 7.2 mg L^{-1}, which exceed Grade V in the Environmental Quality Standards for Surface Water in China [17]. The concentration of NO_3-N of the inorganic nitrogen was the highest, ranging from 1.77 to 2.02 mg L^{-1}, followed by the concentrations of NH_4-N and NO_2-N with ranges from 0.03 to 1.16 mg L^{-1} and 0.03 to 0.20 mg L^{-1}, respectively (Table 1).

Nitrogen pollution in the sediments showed an increasing trend from the upstream to the downstream samples. The concentrations of TN increased from 942.91 mg kg^{-1} at Site J5 to 3,457.63 mg kg^{-1} at Site J1, and the concentration of NH_4-N increased from 48.88 mg kg^{-1} at Site J5 to 270.92 mg kg^{-1} at Site J1. The concentrations of NO_3-N and NO_2-N were also the highest at the downstream Site J1 with the NO_3-N concentration of 112.7 mg kg^{-1} (Table 2).

This study was located in the upstream areas in the northwest region of the Taihu basin, which are the most heavily N-polluted areas [18]. Consequently, the N concentrations in both the stream water and in the sediments showed very high concentrations. Additionally, the concentrations

Table 2
Concentrations of nitrogen forms in the sediments of the five sampling sites in the Hangzhou–Beijing Grand Canal

Site	TN (mg kg^{-1})	NO_3^- (mg kg^{-1})	NO_2^- (mg kg^{-1})	NH_4^+ (mg kg^{-1})
J1	3,457.63	112.74	0.52	270.92
J2	2,122.25	24.20	0.11	104.98
J3	1,596.57	15.94	0.04	54.71
J4	845.27	11.12	0.14	36.14
J5	942.91	22.46	0.06	48.88

Table 1
Overlying water quality parameters at the five sampling sites of the Hangzhou–Beijing Grand Canal

Site	Temperature (°C)	pH	DO (mg L^{-1})	TSS (mg L^{-1})	COD_{Mn} (mg L^{-1})	TN (mg L^{-1})	NH_4-N (mg L^{-1})	NO_2-N (mg L^{-1})	NO_3-N (mg L^{-1})
J1	8.4	8.5	10.0	69	5.5	4.3	0.60	0.04	1.78
J2	8.0	8.3	9.9	121	6.7	7.2	1.16	0.20	1.77
J3	8.2	9.0	9.9	115	4.3	3.3	0.03	0.03	1.78
J4	10.7	8.7	9.8	90	5.0	4.0	0.23	0.08	1.83
J5	14.4	8.8	9.5	111	3.5	4.6	0.52	0.05	2.02

of TN and its exchangeable fractions in the sediments increased from the upstream to the downstream locations of the Beijing–Hangzhou Grand Canal, which also increased with the degree of urbanization (Fig. 1; Table 2). In particular, the heaviest N pollution occurred in the industrialized and urbanized downstream areas such as Wuxi City and Changzhou City, where N from the sewage of urbanized towns and cities substantially increased the N load in the rivers flowing through these areas.

3.2. Sediment properties with particle size distribution

The median diameter of the raw sediment samples was 25.0 μm (Fig. 2). The median diameters of the five size groups were 79.08, 33.73, 26.98, 9.02 and 3.66 μm, all of which were located in the theoretical ranges calculated by the Stokes sedimentation formula. The total mass percentages of the groups with ranges of <5, 5–10, 10–30, 30–50 and 50–150 μm were 5.3%, 15.6%, 25.4%, 26.9% and 26.8%, respectively.

The concentrations of TN and OM in the sediments increased with the decreased particle sizes except for the 50–150 μm particles (Table 3), which were found to contain coarse organic matter during the sieving processes. The concentration of TN in the particles of <5 μm is 1,748.3 mg kg^{-1}, which is almost five times that of the particles of the 30–50 μm size range, whose TN concentration is 346.9 mg kg^{-1}. The fine particles usually have a large specific surface area-to-volume ratio that is responsible for a strong sorption ability for organic matter that contains N in organic forms [6,19]. Additionally, fine particles with a high N concentration could transport and settle downstream during frequent resuspension, resulting in an elevated N concentration in both the sediments and water (Tables 2 and 3). Therefore, the fine particles of <10 μm in sediments, accounting for 20.9% of the raw sediments, could play an important role in N transport in the river due to their enrichment of N. In contrast, the inorganic nitrogen fractions did not show a clear regularity with particle size distribution, especially for NH_4-N. This result could imply that the adsorbed NH_4-N had been nitrified or denitrified on fine particulate matter. Canals are frequently disturbed by shipping hydrodynamics and undergo aeration of stream water (Table 1), which is favourable for nitrification. Some studies confirmed that nitrification on fine particulates was much higher than on coarse particulates in suspended particles [1,20], thus leading to low concentrations of exchangeable NH_4-N and the high concentrations of NO_3-N in the fine particles (Table 3).

3.3. Sorption kinetics and isotherms of NH_4-N on sediments with particle size distribution

The NH_4-N sorption kinetics for the five particle size groups is suitable for the first-order kinetic equation (Fig. 3). The Q_e values of the fine particles were higher than those of the coarse particles, with values of 65.3 to 90.3 mg·kg^{-1} for particles of <5 μm and 5–10 μm vs. 36.6–41.2 mg kg^{-1} for the other three particle size groups. Additionally, the sorption rate constant (k_1) indicated that the particles of <5 μm could reached sorption equilibrium within 1.4 min, which was faster than that of other particle size groups (Table 4).

A linear equation is suitable for the isotherm adsorption of NH_4-N on sediments of the five particle size groups (Fig. 4). The NH_4-N adsorption abilities of the fine particles (those <10 μm) were higher than those of coarse particles (>10 μm).

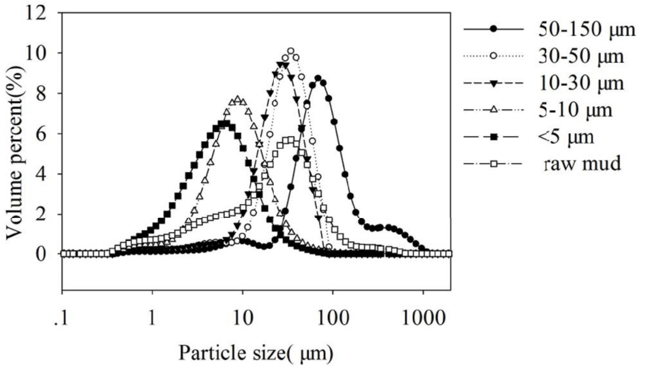

Fig. 2. Grain size distribution of the five particle size groups that were separated using the settling method.

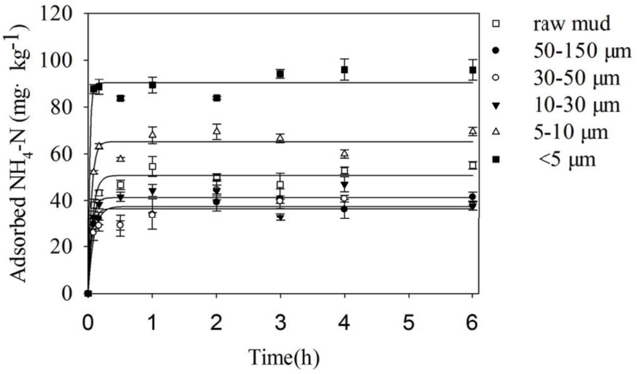

Fig. 3. Adsorption kinetic curves of ammonium nitrogen by sediment particle size.

Table 3
Physical and chemical properties for the five particle size groups

Samples (μm)	d_{50} (μm)	TN (mg kg^{-1})	NO_3^- (mg kg^{-1})	NO_2^- (mg kg^{-1})	NH_4^+ (mg kg^{-1})	OM (%)
50–150	79.08	413.7 ± 4.76	2.8 ± 0.16	0.00 ± 0.00	11.7 ± 0.08	0.76
30–50	33.73	346.9 ± 19.9	2.9 ± 0.06	0.16 ± 0.11	7.0 ± 0.1	0.31
10–30	26.98	520.0 ± 40.3	2.4 ± 0.01	0.03 ± 0.00	5.2 ± 0.12	0.60
5–10	9.02	1,319.8 ± 4.7	2.4 ± 0.01	0.03 ± 0.00	12.0 ± 0.06	1.40
<5	3.66	1,748.3 ± 9.04	18.6 ± 1.22	0.29 ± 0.18	3.6 ± 0.06	2.67

Table 4
Pseudo-first-order kinetic parameters for the ammonium adsorption on the sediments of the five particle size groups and raw mud

Samples (μm)	Q_0 (mg kg^{-1})	k_1 (h^{-1})	R^2	P
Raw mud	50.7	15.0	0.95	0.000**
50–150	36.6	18.0	0.88	0.000**
30–50	37.5	11.6	0.84	0.000**
10–30	41.2	18.6	0.89	0.000**
5–10	65.3	19.4	0.95	0.000**
<5	90.3	42.2	0.97	0.000**

Note: **indicates a significant correlation at the 99% confidence level.

The k_e values were 80.21 L kg^{-1} particles and 54.87 L kg^{-1} for particles of <5 and 5–10 μm, respectively, while those values ranged from 25.39 to 31.80 L kg^{-1} for the coarse particles of >10 μm (Table 5).

The ENC$_0$ values of the coarse particles range from 0.418 to 0.460 mg L^{-1}, which are significantly higher than those of the fine particles (<10 μm) with a range from 0.029 to 0.112 mg L^{-1}. The ENC$_0$ value determines the critical value of the adsorption–desorption equilibrium of the sediments. When the fine particles are suspended in the water, they will adsorb more NH_4-N due to their lower ENC$_0$ values and longer residence time in the water. The fine suspended particles could play the role of a "sink" in stream water by reducing the NH_4-N concentration. In contrast, the coarse particles with higher ENC$_0$ values could possibly play the role of a "source" by releasing more NH_4-N to the overlying water. Some researchers have observed that the NH_4-N released from the sediments was re-adsorbed during the resuspension of the sediments [21,22]. This could be a comprehensive result involving a series of processes, including pore water releasing NH_4-N through ammonification under anoxia and redistribution of NH_4-N between the coarse particles and the fine particles. The specific processes and control mechanisms need to be further clarified.

3.4. Factors affecting NH_4-N adsorption on sediments

Correlation analysis shows that the adsorption affinity energy (k_e) of the particles to NH_4-N was positively correlated to OM and TN, while the ENC$_0$ was negatively correlated to OM and TN. There was a positive correlation between k_1 and concentrations of OM and NO_3-N, as well as between Q_e and the concentrations of OM and TN. In contrast, the sorption parameters were not correlated with the concentration of NH_4-N (Table 6). During the adsorption process, NH_4^+ first aggregates around the particles and then undergoes ligand exchange with the adsorption sites at a fast rate. As NH_4^+ is continually adsorbed, the number of adsorption sites decreases [23]. This research showed that the organic matter of the sediment existed as humus and promoted NH_4-N adsorption as reported in a previous study [24], although some scholars reported that organic acids of small molecules will block NH_4-N adsorption sites [25]. The organic matter in humus provides the adsorption affinity for NH_4-N by a series of functional groups. Fourier transform infrared spectroscopy showed that NH_4-N adsorption could be associated with non-associative acyl groups, ketones, aliphatic methyl and methylene groups [26]. Meanwhile, a complex of the particulate organic matter with alkaline water will generate negative ions to adsorb positively charged NH_4^+. For the fractionation by particle size, some research indicated that the coarse particles are dominated by particulate organic matter while the fine particles tended to adsorb more soluble organic matter containing abundant functional groups for the adsorption of NH_4-N [27]. This finding could explain the difference in adsorption abilities of the different particle sizes.

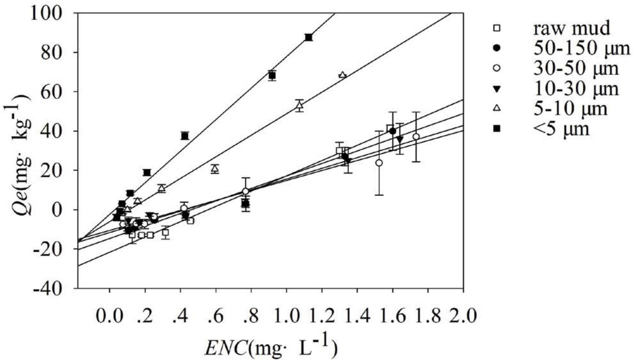

Fig. 4. Isotherms of ammonium on the sediments of the five particle size groups and raw mud.

This research could provide more detailed information about the rivers with frequent resuspension of sediments and with high concentrations of N in the stream water. Fine particles potentially play an important role in nutrient transport due to their strong sorption ability and active biological

Table 5
Adsorption isotherm equation parameters for the five size groups of particles and raw mud

Samples (μm)	Adsorption isotherm equation	k_e (L kg^{-1})	b (mg kg^{-1})	R^2	ENC_0 (mg L^{-1})	P
Raw mud	$y = 38.84x–21.65$	38.84	21.65	0.984	0.557	0.000**
50–150	$y = 31.80x–14.64$	31.80	14.64	0.965	0.460	0.000**
30–50	$y = 25.39x–10.60$	25.39	10.60	0.977	0.418	0.000**
10–30	$y = 27.24x–11.66$	27.24	11.66	0.954	0.428	0.000**
5–10	$y = 54.87x–6.12$	54.87	6.12	0.990	0.112	0.000**
<5	$y = 80.21x–2.36$	80.21	2.36	0.985	0.029	0.000**

Note: **indicates a significant correlation at the 99% confidence level.

Table 6
Correlation analysis between the chemical properties and the adsorption parameters of the five particle size groups

–	k_e	B	ENC_0	k_1	Q_e	OM	NH_4-N	NO_3-N	TN
k_e	1	–	–	–	–	–	–	–	–
b	–0.912*	1	–	–	–	–	–	–	–
ENC_0	–0.954*	0.961**	1.	–	–	–	–	–	–
k_1	0.910*	–0.770	–0.764	1	–	–	–	–	–
Q_e	0.989**	–0.955*	–0.966**	0.902*	1	–	–	–	–
OM	0.990**	–0.869	–0.906*	0.957*	0.975**	1	–	–	–
NH_4-N	–0.266	0.399	0.169	–0.527	–0.354	–0.331	1	–	–
NO_3-N	0.856	–0.765	–0.702	0.958*	0.854	0.897*	–0.632	1	–
TN	0.981**	–0.943*	–0.986**	0.849	0.988**	0.956*	–0.234	0.769	1

*Significant correlation at the 95% confidence level.
**Significant correlation at the 99% confidence level.

processes [28]. The functions of the particles on N transformation need to be further clarified in the turbid water with heavy N pollution.

4. Conclusions

This research indicated that the heterogeneity of the sediments causes differences in the N composition of the different particle size groups, providing an insight into the understanding of N transport with more details. The main conclusions include the following:

- The N concentrations of the five particle size groups range from 346.9 to 1,748.3 mg/kg, with increased TN in the smaller particle sizes.
- The fine particles showed stronger ammonium adsorption abilities compared with those of the coarse particles, and this phenomenon is consistent with the organic matter concentrations. However, ammonium cannot be retained on the fine particles due to active biological transformations.

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Symbols

Q_e — Sorbed nitrogen at the equilibrium states, mg·kg^{-1}
ENC — Equilibrium nitrogen concentration, mg·L^{-1}
ENC_0 — Zero-adsorption equilibrium nitrogen concentration, mg·L^{-1}
k_e — Nitrogen binding energy constant, L·kg^{-1}
b — Native adsorbed nitrogen, mg·kg^{-1}
k_1 — First-order constant of sorption kinetics, h^{-1}
Q_t — Amount of sorbed nitrogen at time t, mg·kg^{-1}
t — Time, h

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