



Superposition effect of floating and fixed beds in series for enhancing nitrogen and phosphorus removal in a multistage pond system

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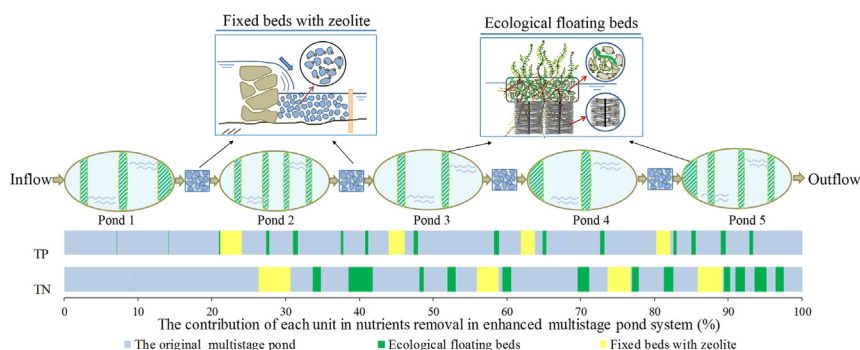
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HIGHLIGHTS

- Floating/fixed beds were integrated into a pond system to enhance nutrients removal.
- A tank-in-series model was established for evaluating the removal efficiency.
- The pond system modification increased TN and TP removals by 12.2 and 13.9%.
- Adsorption and/or denitrification were identified to be the main actions performed.

GRAPHICAL ABSTRACT



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ABSTRACT

In order to improve the efficiency of nitrogen and phosphorus removal in a multistage pond system which receives polluted natural inflow and outflows to a landscape lake, ecological floating beds (EFBs) were installed along the flow-path of each pond and fixed beds (FBs) were embedded in between each pair of ponds. Such a simple modification of the MPS effectively enhanced the total nitrogen (TN) removal rate from 59.2% to 71.4% and the total phosphorus (TP) removal rate from 37.1% to 51.0%. It was identified that the EFBs mainly contributed to enhanced TN removal by the biomass growth in the stereo-elastic packing and attachment on the surface of ceramsite particles packed in the floating mat, while the FB filled with zeolites contributed to both TP adsorption and biological TN removal to certain extent, as indicated by the denitrification rate and adsorption function experimentally obtained for each part of the bed settings. The superposition effect of the installation of EFBs and FBs was estimated using a tank-in-series model. With a Nash-Sutcliffe efficiency higher than 0.75, calculation results of the model well fitted field measurements and showed that the EFBs (including plant uptake) contributed to the increase of TN and TP removal by 23.3% and 8.12%, respectively, and that contributed by FBs were 19.6% and 10.7%, respectively.

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1. Introduction

Urban ponds are important components of the built environment because of their ornamental and recreational function (Waaen et al., 2016a). Ponds have been used extensively across the world for treating agricultural, industrial and municipal wastewater due to their low cost and effective removal of pathogens, organic pollutants and nutrient (Ragush et al., 2017; Zimmo et al., 2004). A pond can be conceptualized as a self-sufficient treatment unit that treats sewage with natural purification processes, driven by the influence of temperature and sunlight (Alhashimi and Hussain, 2013; Butler et al., 2015; Gruchlik et al., 2018). However, due to excessive phosphorous (P) and nitrogen (N) inputs and the limits of self-purification, eutrophication has become common in many urban ponds and has severe impacts on water quality (Waaen et al., 2016b).

Recently, different strategies including ex-situ and in-situ remediation technologies have been used to remove phosphorous and nitrogen for eutrophication control (Hasan et al., 2019; Yi et al., 2009a). Blast furnace slag filters have been found to improve phosphorous removal from the effluent in existing pond systems and have the potential to produce a final effluent that meets acceptable levels of P discharge (for example, <2 mgP/L) (Valero et al., 2012). Constructed wetlands (CWs) have been extensively applied for the treatment of landscape water in many countries due to their advantages of versatile pollutant removal performance and good landscape integration (Al-Rubaei et al., 2017; Tsalkatidou et al., 2009; Wang et al., 2015). Even so, compared with ex-situ remediation, which requires additional land area and complex construction, in-situ technology may be more suitable for ponds. The ecological floating bed (EFB) is a good choice for nutrient removal in landscape water (Li et al., 2010; Olguin et al., 2017). Wang et al. (2018) used an EFB with *calamus*, *iris*, *lythrum* and *Hydrocotyle vulgaris* and a strengthened EFB with zeolite and sponge iron as fillers to reduce the nitrogen content in urban landscape water. However, the use of only individual treatment processes is insufficient for the enhancement of nutrient removal in ponds and integrative treatment technology may be the development trend of pond enhancement in the future. Moreover, compared with large treatment facilities which require additional land area and complex construction, a series of small in-situ remediation facilities may be more conducive to maintaining the original appearance of ponds. In addition, their superposition effect can also be expected.

The mechanism for phosphorous removal in ponds principally comprises chemical precipitation and various biological treatment processes such as uptake by algae or plants (Picot et al., 1991; Surampalli et al., 1995). There are also many mechanisms for nitrogen removal in ponds such as ammonia volatilization, plant uptake and denitrification (Mukhtar et al., 2017). In recent years, various models have been used to analyse the potential removal of phosphorous and nitrogen, including the first-order model and the mechanistic model. Bastos et al. (2014) deduced a function of the ammonium surface loading rate using the first-order model and used it to predict ammonium removal in facultative and maturation ponds. However, much of the previous research has focused on the migration of phosphorous and nitrogen in ponds using these models (Houng and Gloyna, 1984; Mukhtar et al., 2017). There is no quantitative description of the proportional contribution of these mechanisms, which is not beneficial for the purposeful selection of technical measures for eutrophication control. Moreover, although the performance of some technological developments has been confirmed in many papers, there exists little quantitative description of the performance of actual mechanisms at work in each improvement technology, especially in integration technologies. A quantitative calculation of these mechanisms of nutrient removal in each technology is conducive for the further study of integration technologies and to reasonably evaluate the effects of various mechanisms on enhancement.

Several small areas of EFBs and fixed beds (FBs) with zeolites were built in the spaces within and in-between the existing ponds to increase nutrient removal in a multistage pond system (MPS). The objectives of

this study are (1) to evaluate the superposition effect of these EFBs and FBs, and (2) to quantify the contribution of the mechanism of each EFB and FB in the nutrient removal of ponds via the tank-in-series model which is based on first-order kinetics and the assumption of completely mixed conditions. The results from this study will offer a reference for the enhanced phosphorous and nitrogen removal in pond systems, and the utilization of the space within and in-between the existing ponds.

2. Materials and methods

2.1. Case explanation

The study was carried out in a five-stage pond system (118°21'E-120°30'E, 29°11'N-30°33'N) with a length of 2.8 km, which is one of the main upstream stretches of West Lake. The pond system contains an ecological corridor with a total length of 950 m. Many different plants are grown in this MPS such as *Nymphaea tetragona* and *Ecklonia stolonifera*. These plants are harvested in November every year. The area of each pond (Ponds 1–5) is 3674, 5745, 4677, 2042 and 13,652 m² with the average depths of 0.9, 1.2, 0.9, 0.9 and 0.9 m, respectively. The flow rate of the MPS is about 2592 m³/d and the hydraulic retention time (HRT) is 11 d. Due to agricultural non-point pollution, the nitrogen concentration of MPS fail to meet landscape water quality standards. The average annual concentration of inflow total nitrogen (TN) is 5.92 mg/L, of which the nitrate (NO₃-N) concentration is 5.60 mg/L, the ammonia nitrogen (NH₄-N) concentration is 0.30 mg/L and the nitrite nitrogen (NO₂-N) concentration is 0.01 mg/L. The average annual concentration of inflow total phosphorous (TP) is 0.041 mg/L, of which the phosphate (PO₄-P) concentration is 0.040 mg/L. According to "Environmental quality standard of surface water" in China, the limit allowable TN and TP concentrations (namely Class V standard) has been set as 2.0 and 0.2 mg/L, respectively, which are taken as the minimum target of water quality improvement in this study.

To improve the efficiency of nitrogen and phosphorous removal in the MPS, EFBs were installed along the flow-path of each pond and FBs were built in between each pair of ponds. The structure of the EFBs and FBs are depicted in Fig. 1(a). Each EFB was divided into three layers, an upper layer planted with *Myriophyllum verticillatum*, a buoyant layer filled by ceramic pellets, and a lower layer hung with stereo-elastic packing. The height of the buoyant layer was 20–25 cm and the diameters of the ceramic pellets were 3–6 cm. The length of the stereo-elastic packing was determined by the water depth, usually 50 cm, and the diameter was 12 cm. The FBs were primarily composed of zeolites with diameters of 2–4 cm. The depths of the FBs were 60 cm. The surface shape of the FBs was generally trapezoidal, with a height of 3–8 m and a bottom side of 8–12 m, which depended on the terrain of the inlets of the ponds. The construction of the EFBs and FBs took place between September and October 2014. The sizes of the EFBs and FBs varied according to the stream geometry and the total combined areas of the FBs and EFBs were 332.1 and 876 m², respectively.

2.2. Field experiments

2.2.1. Water sampling

Water samples were collected in two stages. We first took water samples from the MPS in its original state, September 2013 and August 2014 before the construction of EFBs and FBs between. After the construction of EFBs and FBs, we took samples from the same locations of the MPS in its enhanced state from September 2016 to August 2017. During each stage, the inflow and outflow of each pond were sampled monthly in the morning with 500 mL glass bottles. The pH, dissolved oxygen (DO), and temperature were measured on-site using a HORIBA U-50 series multi-parameter water quality checker. All the water samples were transported to the laboratory within 6 h of collection for chemical analyses.

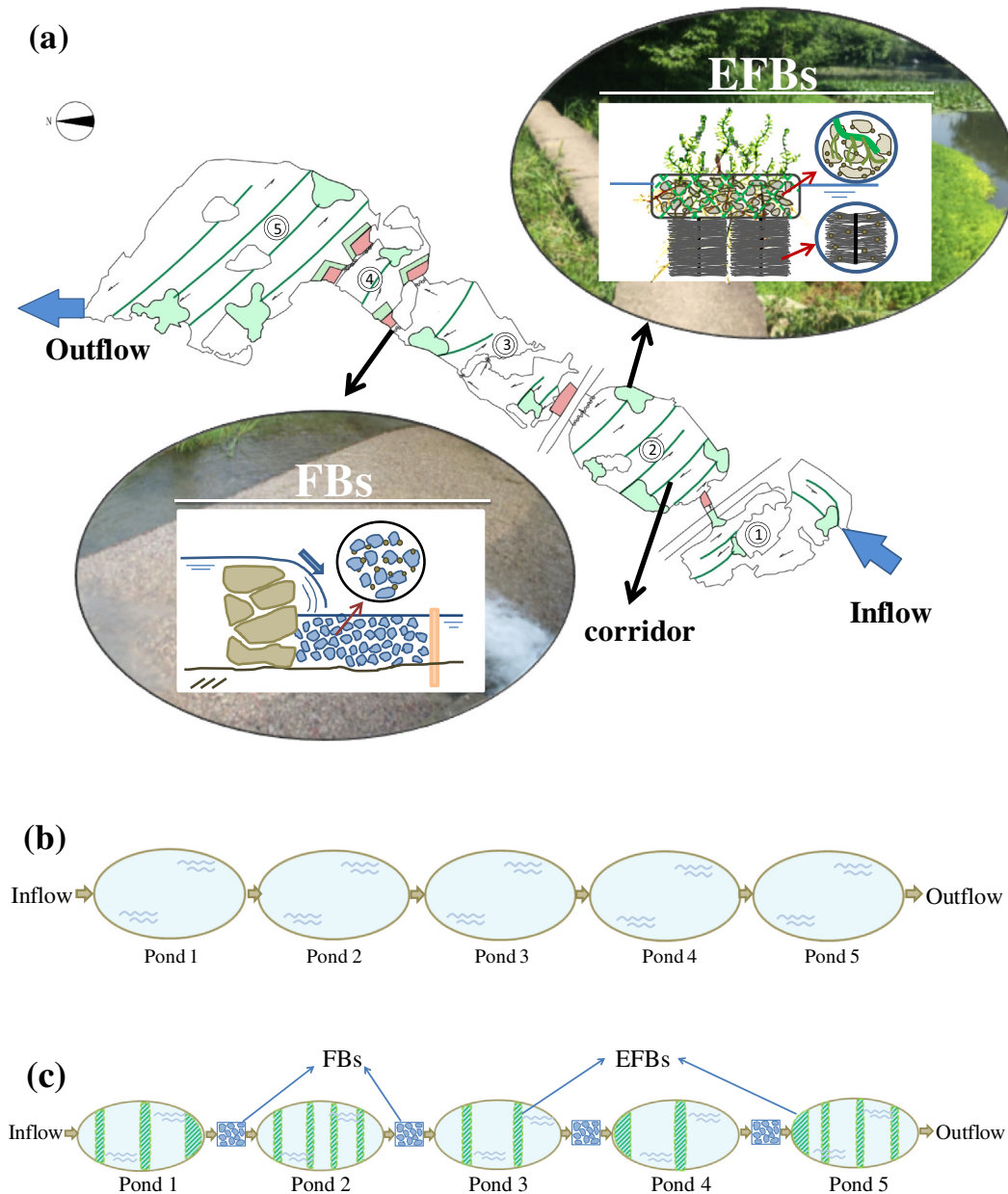


Fig. 1. The schematic diagram of MPS. (a) the topographic diagram of the MPS and the distribution and design of EFBs and FBs; (b) the model diagram of original MPS; (c) the model diagram of enhance MPS.

2.2.2. Plant sampling

Plant uptake is an important part of P and N removal in MPS due to the plant harvest in November. Four plant species that have always existed in the MPS were selected for sampling due to their abundance: *Nymphaea tetragona*, *Nuphar pumila*, *Thalia dealbata* and *Ecklonia stolonifera*. Because these plants were planted in the ponds long before 2014, the biomass is stable (Yucong Zheng et al., 2018). Moreover, according to a message from the harvest unit, the biomasses of different plants were similar in every year from 2014 to 2017, and it can therefore be assumed that the removal amount of the four plants in both the original and enhanced MPS was the same. The *Myriophyllum verticillatum* species on the EFBs in the enhanced MPS was also sampled.

The number, weight and coverage of each species were measured and three 0.25 m² plant biomasses of different plants were randomly selected before harvesting in November. The harvested plants were separated into leaves, stems and flowers and were washed with distilled

water to remove the adhering water and sediments. The samples were then oven-dried at 80 °C for at least 48 h to achieve a constant weight. The dried plant materials were ground into powder in a grinding machine and filtered through a 0.25 mm mesh sieve (Zheng et al., 2016). The samples were then transferred in identical quantities into destruction tubes to digest the contents in a block using a concentrated sulphuric-salicylic mixture with selenium as a catalyst. The TN and TP contents were then analysed via the Kjeldahl method and molybdenum blue method, respectively (Bao, 2000). The amount of nutrient uptake by each plant was calculated according to the following equation:

$$m_{total} = (M_{leaves} \times C_{leaves}) + (M_{stem} \times C_{stem}) + (M_{flowers} \times C_{flowers}) \quad (1)$$

where M represents the total biomass of leaves, stems and flowers (g), and C represents the average concentration of nitrogen and phosphorus in the respective plant parts (mg/g).

2.3. Laboratory experiments

2.3.1. Experimental setting

Adsorption experiments of $\text{NH}_4\text{-N}$ and $\text{PO}_4\text{-P}$ were conducted by equilibrating zeolites in a glass tube.

Denitrification experiments were conducted on carriers from the enhanced MPS in 1 L Erlenmeyer flasks containing 800 mL of nutrient solution. The nutrient solution (1 L) was composed of the following: 377 mg glucose, 400 mg KNO_3 , 0.5 mg NaH_2PO_4 , 72 mg KCl , 180 mg $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, 10.6 mg CaCl_2 , 225 mg NaHCO_3 and 1.2 mL microelement. The microelement (1 L) was composed as follows: 375 mg $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, 30 mg $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$, 0.5 mg H_3BO_3 , 30 mg $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$, 72 mg $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, 10.6 mg KI and 2500 mg EDTA . All chemicals used were of analytical grade.

2.3.2. Adsorption experiments

Adsorption experiments were conducted to determine the parameters of zeolite adsorption. $\text{NH}_4\text{-N}$ adsorption experiments were conducted with 0.2 g of zeolites (20–40 mesh) in triplicate using a 20 mL $\text{NH}_4\text{-N}$ solution of varied initial concentrations (50, 70, 90, 110, 130, 150, 170 and 190 mg/L). The zeolites were sampled from the enhanced MPS in August 2017 and transported to the laboratory immediately where they were washed with distilled water and dried at 30 °C for 4 h. The dried zeolites were ground and filtered through 20-mesh and 40-mesh sieves and 20–40 mesh zeolite was obtained. The solutions with zeolite were stirred continuously at 150 rpm in a horizontal shaker for 24 h at different temperatures (10, 20 and 30 °C). After 24 h, the samples were centrifuged at 2000 rpm and analysed to determine the residual $\text{NH}_4\text{-N}$ concentrations. The same process was carried out for the adsorption experiment of $\text{PO}_4\text{-P}$. The amounts of $\text{NH}_4\text{-N}$ and $\text{PO}_4\text{-P}$ adsorbed by the zeolites were calculated using Eq. (2). The equilibrium of adsorption was evaluated using the Freundlich isotherm model:

$$q_e = \frac{V(C_0 - C_e)}{m} \quad (2)$$

$$\ln q_e = \frac{1}{n} \ln C_e + \ln K_f \quad (3)$$

where C_0 is the initial concentration (mg/L), C_e is the equilibrium concentration (mg/L), q_e is the amount of adsorbed molecule (mg/g), V is the volume of each solution (mL), m is the total amount of zeolite (g), K_f is the Freundlich constant related to the adsorption capacity of the adsorbent, and $1/n$ is the Freundlich exponent related to surface heterogeneity.

2.3.3. Denitrification experiments

Denitrification experiments of the carriers were conducted to calculate the denitrification rates of the EFBs and FBs. The denitrification experiment was carried out as described by Xu et al. (2013). First, the carriers (the ceramic pellets, stereo-elastic packing and zeolites) were transferred from the EFBs and FBs in the enhanced MPS into Erlenmeyer flasks and washed with distilled water three times to remove the residual nitrogen compounds and organic matter. Then, 800 mL nutrient solutions were added to the flasks. The flasks were sealed and incubated at ambient temperature, and 10 mL of the solution was sampled from each flask to analyse the COD, TN, $\text{NO}_3\text{-N}$ and $\text{NO}_2\text{-N}$ once every 12 h. The masses of the ceramic pellets, stereo-elastic packing and zeolite used in the batch experiments were about 70, 3 and 120 g respectively. Each experiment was carried out in triplicate. Due to the influence of temperature, the denitrification experiments of the three carriers were conducted in different seasons to determine the denitrification rate at 20 °C. According to the changes in $\text{NO}_x\text{-N}$ ($\text{NO}_3\text{-N} + 0.6\text{NO}_2\text{-N}$), the denitrification rates of various carriers were calculated by the

following equation (Sage et al., 2006):

$$V_D = \frac{d(C_{\text{NO}_x} - N)}{Xdt} \quad (4)$$

$$V_D = V_{D20}\theta^{(T-20)} \quad (5)$$

where, V_{DN} is the denitrification rate (mg N/(g·h)), X is the wet weight of the carrier (g), and θ is the temperature correction coefficient.

The results of batch experiments were fitted into both zero- and first-order kinetic models to study the nitrogen removal processes of the carriers according to Eqs. (13) and (14), respectively (Rout et al., 2016)

$$C_t = C_0 - K_0 t \quad (6)$$

$$C_t = C_0 e^{-kt} \quad (7)$$

where C_0 and C_t are the concentrations of TN at time 0 and time t (mg/L), respectively, t is the time of denitrification (h), K_0 is the zero-order rate constant (mg/L/h), and K is the first-order rate constant (1/h).

2.3.4. Chemical analysis

The parameters including TN, $\text{NO}_3\text{-N}$, $\text{NH}_4\text{-N}$, $\text{NO}_2\text{-N}$ and TP were measured and all chemical analyses were carried out according to Standard methods (APHA, 1998). The TN was determined by the alkaline potassium persulfate digestion ultraviolet spectrophotometer method, and the TP was measured according to molybdenum antimony spectrophotometry. We detected $\text{NH}_4\text{-N}$ using Nessler's reagent spectrophotometry, $\text{NO}_2\text{-N}$ was detected by N-(1-naphthyl) ethylenediamine dihydrochloride, COD was measured by the permanganate method, and $\text{NO}_3\text{-N}$ was determined in 0.45- μm filtered samples by ion chromatography with conductivity detection (Xiong et al., 2016).

2.4. Mathematical modeling

2.4.1. Tank-in-series model

As the ponds in the original MPS were in series, the tank-in-series model was used to estimate the nitrogen and phosphorus removal effects. There were five tanks in the original MPS (Fig. 1(b)). Each unit in the tank-in-series model was based on first-order kinetics and under the assumption of completely mixed conditions.

In the MPS, 95% of P existed in the form of $\text{PO}_4\text{-P}$. To simplify the calculation, organic-P was ignored. The removal of TP in the ponds is described by Eq. (8). The MPS contained three kinds of nitrogen: organic-N, $\text{NH}_4\text{-N}$ and $\text{NO}_x\text{-N}$. Because $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ accounted for over 95% of the N content, organic-N and $\text{NO}_2\text{-N}$ were ignored. Ammonia was removed via nitrification and plant uptake. Nitrification and denitrification were modeled using a first-order decay expression. The effect of T on the nitrification and denitrification rates was evaluated using the Arrhenius equation:

$$\frac{dC_P}{dt} = k_P C_P + r_P \quad (8)$$

$$\frac{dC_{\text{TN}}}{dt} = \frac{dC_{\text{NH}}}{dt} + \frac{dC_{\text{NO}}}{dt} + r_N \quad (9)$$

$$\frac{dC_{\text{NH}}}{dt} = k_n \theta^{(T-20)} C_{\text{NH}} \quad (10)$$

$$\frac{dC_{\text{NO}}}{dt} = k_d \theta^{(T-20)} C_{\text{NO}} - k_n \theta^{(T-20)} C_{\text{NH}} \quad (11)$$

where C_{TP} , C_{TN} , C_{NH} and C_{NO} are the concentrations of TP, TN, $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$, respectively (mg/L), t is the HRT (d), k_{TP} is the first-order removal rate of TP (d^{-1}), k_n is the nitrification rate of $\text{NH}_4\text{-N}$ at 20 °C

(d^{-1}), k_d is the denitrification rate of NO_3-N at $20^\circ C$ (d^{-1}), θ is the temperature correction coefficient (1.01–1.09 for ponds) (Mukhtar et al., 2017), and r_p and r_n are the plant uptake of phosphorus and nitrogen in ponds (mg/L/d) and are presented as mg-P and mg-N per day per volume of the pond, respectively. According to the plants experiment, the TN contents in the *Nymphaea tetragona*, *Nuphar pumila*, *Thalia dealbata* and *Ecklonia stolonifera* species were 3.05, 4.91, 4.06 and 2.18%, respectively, and the TP contents were 2.63, 4.08, 2.87 and 4.61 g/kg, respectively.

Based on the TN and TP concentrations of inflow and outflow of each pond from September 2013 to August 2014, the values of k_p , k_n , and k_d of each pond were calculated. The calculation process is as follows:

For the TP in Pond i, according to Eq. (1),

$$k_{pi} = \frac{C_{p0i} - C_{pi}}{t_i C_{p1}} - \frac{r_{pi}}{C_{pi}} \quad (12)$$

where C_{p0i} and C_{pi} are the observed TP concentrations of the inflow and outflow of Pond i, respectively, t_i is the HRT of Pond i, and k_{pi} is the k_p value of Pond i.

For the TN in Pond i, the calculation of k_{ni} is similar to that of k_{pi} .

$$k_{ni} = \frac{C_{NH0i} - C_{NHi}}{t_i \theta^{(T_i-20)} C_{NHi}} - \frac{r_{Ni}}{C_{NHi}} \quad (13)$$

where C_{NH0i} and C_{NHi} are the observed NH_4-N concentrations of the inflow and outflow of Pond i, respectively, k_{ni} is the k_n value of Pond i, and T_i is the temperature of Pond i.

The calculation of k_{di} is as follows:

$$k_{di} = \frac{C_{NO0i} + k_{n1} \theta^{(T_i-20)} C_{NHi} - C_{NOi}}{t_i \theta^{(T-20)} C_{NOi}} \quad (14)$$

where C_{NO0i} and C_{NOi} are the observed NO_3-N concentrations of the inflow and outflow of Pond i, respectively, and k_{di} is the k_d value of Pond i.

To calibrate and evaluate the tank-in-series model, the Nash-Sutcliffe efficiency (NSE) was used with the observed data, with dimensionless parameters scaled onto the interval [−inf to 1.0] (Gupta et al., 2009). An NSE value of 1.0 represents a perfect match and a model with an NSE value of 0.0 is no more accurate than the calculation of the mean value (Jain and Sudheer, 2008).

$$NSE = 1 - \frac{\sum_{i=1}^n (Y_i - \bar{Y}_i)^2}{\sum_{i=1}^n (Y_i - \bar{Y})^2} \quad (15)$$

where Y_i and $\bar{Y}_i$ are the calculated and measured values of the criterion variable Y , respectively, $\bar{Y}$ is the mean of the measured values of Y , and n is the sample size.

The calculation processes for the calculated TP and TN in each pond are as follows (the inflow of Pond i is the outflow of Pond i-1).

For TP:

$$C_{p1} = \frac{C_{p0} - r_{p1} t_1}{1 + k_{p1} t_1} \quad (16)$$

$$C_{p2} = \frac{C_{p1} - r_{p2} t_2}{1 + k_{p2} t_2} = \frac{\frac{C_{p0} - r_{p1} t_1}{1 + k_{p1} t_1} - r_{p2} t_2}{1 + k_{p2} t_2} \quad (17)$$

And so on.

$$C_{p5} = \frac{C_{p4} - r_{p5} t_5}{1 + k_{p5} t_5} \quad (18)$$

The calculation process of calculated NH_4-N is similar to that of calculated TP.

$$C_{NH5} = C_{NH4} \frac{C_{NH4} - r_{Ni} t_5}{1 + k_{n5} \theta^{(T_i-20)} t_5} \quad (19)$$

The calculation process of calculated NO_3-N is as follows:

$$C_{NO1} = \frac{C_{NO0} + k_{n1} \theta^{(T_1-20)} C_{NH1}}{1 + t_1 k_{d2} \theta^{(T_1-20)}} \quad (20)$$

$$C_{NO2} = \frac{C_{NO1} + k_{n2} \theta^{(T_2-20)} C_{NH2}}{1 + t_2 k_{d2} \theta^{(T_2-20)}} \quad (21)$$

$$= \frac{\frac{C_{NO0} + k_{n1} \theta^{(T_1-20)} C_{NH1}}{1 + t_1 k_{d1} \theta^{(T_1-20)}} + k_{n2} \theta^{(T_2-20)} C_{NH2}}{1 + t_2 k_{d2} \theta^{(T_2-20)}}$$

where C_{NOi} is the NO_3-N concentration of the outflow of Pond i and k_{di} is the k_d value of Pond i.

The NO_3-N concentration of each pond was calculated by analogy.

2.4.2. Superposition of EFBs and FBs

The tank-in-series model was also used to estimate the superposition of the EFBs and FBs. In each enhanced pond, because of the added EFBs, the pond was divided into several parts in series, including EFBs and the segmented water body. The FBs were set in between pairs of ponds, and were in series with the ponds. In other words, the EFBs, the segmented pond body and the FBs were connected together in series, and 36 tanks were formed (Fig. 1(c)). The nitrogen and phosphorus removal in the enhanced MPS are described by Eqs. (22)–(25).

$$\frac{dC_p}{dt} = k_p C_p + f(C_0)_{p-adsorption} + r_p + r_{p-plant-bed} \quad (22)$$

$$\frac{dC_{TN}}{dt} = \frac{dC_{NH}}{dt} + \frac{dC_{NO}}{dt} + r_N \quad (23)$$

$$\frac{dC_{NH}}{dt} = k_{n20} \theta^{(T-20)} C_{NH} + r_N + f(C_0)_{N-adsorption} + r_{N-plant-bed} \quad (24)$$

$$\frac{dC_{NO}}{dt} = k_{d20} \theta^{(T-20)} C_{NO} + k_{d-bed20} \theta^{(T-20)} C_{NO} - k_{n20} \theta^{(T-20)} C_{NH} \quad (25)$$

where $f(C_0)_{p-adsorption}$ and $f(C_0)_{N-adsorption}$ are the adsorption functions of zeolites on phosphorus and nitrogen based on adsorption experiments (mg/L/d), respectively, k_d is the denitrification rate of NO_3-N of the EFBs and FBs at $20^\circ C$ (d^{-1}) and is determined by denitrification experiments, $r_{p-plant-bed}$ and $r_{N-plant-bed}$ are the amounts of phosphorus and nitrogen removed by plants on the pond bed (mg/L/d), respectively, and the concentrations of TN and TP in the entire *Myriophyllum verticillatum* plant are 2.97% and 3.14 g/kg, respectively.

The NSE was also utilized to evaluate the tank-in-series model using the EFBs and FBs. The calculated TN and TP concentrations of the outflow of each pond in the enhanced five-stage pond were obtained from the various k values of the original five-stage pond and the parameters of the EFBs and FBs. The calculation process is described in detail, using Pond 1 and FB1 as an example.

Pond 1 and FB1 contained three EFBs. Because the last EFB was at the end of the pond, the pond was divided into three parts, forming a series sequence of segments named water 1, EFB 1, water 2, EFB 2, water 3, EFB 3 and FB1. Each segment of water was regarded as a separate complete mixed reactor.

According to Eq. (5), the TP of the outflow in water 1 was calculated as:

$$C_{Pw1} = \frac{C_{P10} - r_{P1} t_{1w1}}{1 + k_{P1} t_{1w1}} \quad (26)$$

and in EFB 1 as:

$$C_{P1E1} = \frac{C_{P1w1} - r_{P1} t_{1E1} - r_{P-plant-EBF1} t_{1E1}}{1 + k_{P1} t_{1E1}} \quad (27)$$

$$= \frac{\frac{C_{P10} - r_{P1} t_{1w1}}{1 + k_{P1} t_{1w1}} - r_{P1} t_{1E1} - r_{P-plant-EBF1} t_{1E1}}{1 + k_{P1} t_{1E1}}$$

where C_{P1w1} and C_{P1E1} are the TP concentrations of the outflows of water i and EFB i in Pond 1, respectively, C_{P10} is the TP concentration of the inflow of Pond 1, $r_{P-plant-EBF1}$ is the amount of phosphorus removed by the plant uptake of EFB i, and t_{1w1} and t_{1E1} are the detention times of water i and EFB i in Pond 1, respectively.

By analogy, the calculated TP of Pond 1 was obtained and is the outflow of EFB 3.

$$C_{P1E3} = \frac{C_{P1w3} - r_{P1} t_{1E3} - r_{P-plant-EBF3} t_{1E3}}{1 + k_{P1} t_{1E3}} \quad (28)$$

The outflow of Pond 1 is the inflow of FB 1. The mechanism of P removal in FB1 includes adsorption by zeolites and removal by water. Simplifying the calculation, we assume that the self-purification of the pond occurs first, followed by adsorption:

$$C_{PF1} = \frac{C_{P1w3}}{1 + k_{P2} t_{F1}} \quad (29)$$

where C_{PF1} is the TP concentration of the outflow of FB 1 using the action of water, t_{F1} is the detention time of FB 1.

The TP concentration of outflow of FB 1 was calculated by C_{PF1} and $f(C_{PF0})_{1P-adsorption}$, which is the inflow of Pond 2:

$$C_{P20} = f(C_{PF0})_{P-adsorption} \quad (30)$$

By this analogy, the TP of the outflow of each pond can be calculated.

The mechanism of NH_4 -N removal includes adsorption and plant uptake in EFBs and FBs and is therefore similar to the mechanism of TP removal in EFBs and FBs. The calculations for NH_4 -N were therefore similar to those for TP. The calculations for determining NO_3 -N as follows:

In water 1:

$$C_{NO1w1} = \frac{C_{NO10} + t_{1w1} k_n \theta^{(T_1-20)} C_{NH1w1}}{1 + k_{d1} \theta^{(T_1-20)} t_{1w1}} \quad (31)$$

and in EFB 1:

$$C_{NO1E1} = \frac{C_{NO1w1} + t_{1E1} k_n \theta^{(T_1-20)} C_{NH1E1} - t_{E1} k_{d-1E1} \theta_E^{(T_1-20)}}{1 + k_{d1} \theta^{(T_1-20)} t_{1E1}} \quad (32)$$

where C_{NO1w1} and C_{NO1E1} are the NO_3 -N concentrations of the outflows of water i and EFB i in Pond 1, respectively, C_{NO10} is the NO_3 -N concentration of the inflow of Pond 1, and k_{d-E1} is the denitrification rate of EFB i.

By analogy, the calculated NO_3 -N of Pond 1 can obtained and is the outflow of EFB 3.

$$C_{NO1E3} = \frac{C_{NO1w3} + t_{1E3} k_n \theta^{(T_1-20)} C_{NH1E1} - t_{E3} k_{d-1E1} \theta_E^{(T_1-20)}}{1 + k_{d1} \theta^{(T_1-20)} t_{1E3}} \quad (33)$$

NO_3 -N was removed by the FBs via denitrification, the calculation for which is as follows:

$$C_{NOF1} = \frac{C_{NO1E3} + t_{F1} k_n \theta^{(T-20)} C_{NH1E1} - t_{F1} k_{d-F1} \theta_F^{(T-20)}}{1 + k_{d2} \theta^{(T-20)} t_{F1}} \quad (34)$$

where C_{NOF1} and C_{NH1E1} are the NO_3 -N and NH_4 -N concentrations of the outflow of FB1, respectively, and k_{d-F1} is the denitrification rate of FB1.

By this analogy, the NO_3 -N of the outflow of each pond can be calculated.

The removal quantity by each unit of the 36 tanks in series can therefore be known and the superposition effect of the EFBs and FBs can be calculated.

3. Results and discussion

3.1. Evaluation of the original multi-pond system

Due to plant uptake, physicochemical action and microbial action in the original MPS, which was characterized by inflow rates of 0.042 and 7.05 mg/L, the observed TP and TN concentrations of the outflow from Pond 1 to Pond 5 decreased by self-purification, as determined by the field experiments. The TP values for Ponds 1–5 were 0.040, 0.032, 0.028, 0.023, and 0.018 mg/L, and the TN values were 5.95, 5.34, 4.81, 4.51 and 4.24 mg/L, respectively. In addition, the average observed TP and TN removal rates for the entire original MPS were 59.2% and 37.1%, respectively (Fig. S1).

To clarify the contributions of these mechanisms, the tank-in-series model was used to quantify the P and N removal processes. The average calculated TP values of the outflows from Pond 1 to Pond 5 were 0.036, 0.031, 0.027, 0.022 and 0.016 mg/L and the average calculated TN values were 5.95, 5.33, 4.85, 4.57 and 4.33 mg/L, respectively, which were close to the observed values of the field experiments (Fig. 2). Moreover, the NSE values for the TP and TN in the original MPS were 0.75 and 0.83, respectively, indicating that the model revealed accurate details about nutrient removal and transformation processes in the MPS.

From the tank-in-series model and Eq. (8), it was determined that 51.2% of P removal in MPS was carried out by plant adsorption which was therefore the primary mechanism, while 48.8% was contributed by the role of k_{TP} . The P removal in ponds principally involves chemical precipitation and biomass (i.e., algae, bacteria and plant) assimilation in the pond system (Strang and Wareham, 2006; Zhou et al., 2006). If the biomass is not removed from the water, TP removal would not be achieved. Therefore, k_{TP} mainly refers to the role of chemical precipitation. However, in many studies, chemical precipitation has been reported to be a prominent mechanism of P removal (Nairn and Mitsch, 1999; Strang and Wareham, 2006). This may be due to the low phosphorus concentration. Ma et al. (2016) reported that plant uptake contributed to 81.0%–95.1% of P removal in ponds with an initial phosphorus concentration of <0.4 mg/L. Compared with other self-purification mechanisms such as chemical precipitation, P is more easily removed by plant uptake in low phosphorus environments.

For N removal, plant absorption was responsible for 3.8% of the TN removal in the original MPS, while denitrification was responsible for the remaining 96.2%. Due to the high nitrogen concentration, the contribution of plant uptake to nitrogen removal was far less than that to P removal. Plants might therefore be a good choice for water quality purification at low nutrient concentrations. In many studies, denitrification was the main mechanism for nitrogen removal (Camargo Valero et al., 2010; Tang, 1998), and denitrification in this study was more likely to occur in sediment than in water because of the DO, which was 7.3 mg/L. Sediment is the location where denitrification predominantly occurs in ponds (Bastviken et al., 2005; Seitzinger, 1988), because anaerobic and anoxic environments are more easily formed in sediments with a high DO. EFBs and FBs were built to improve

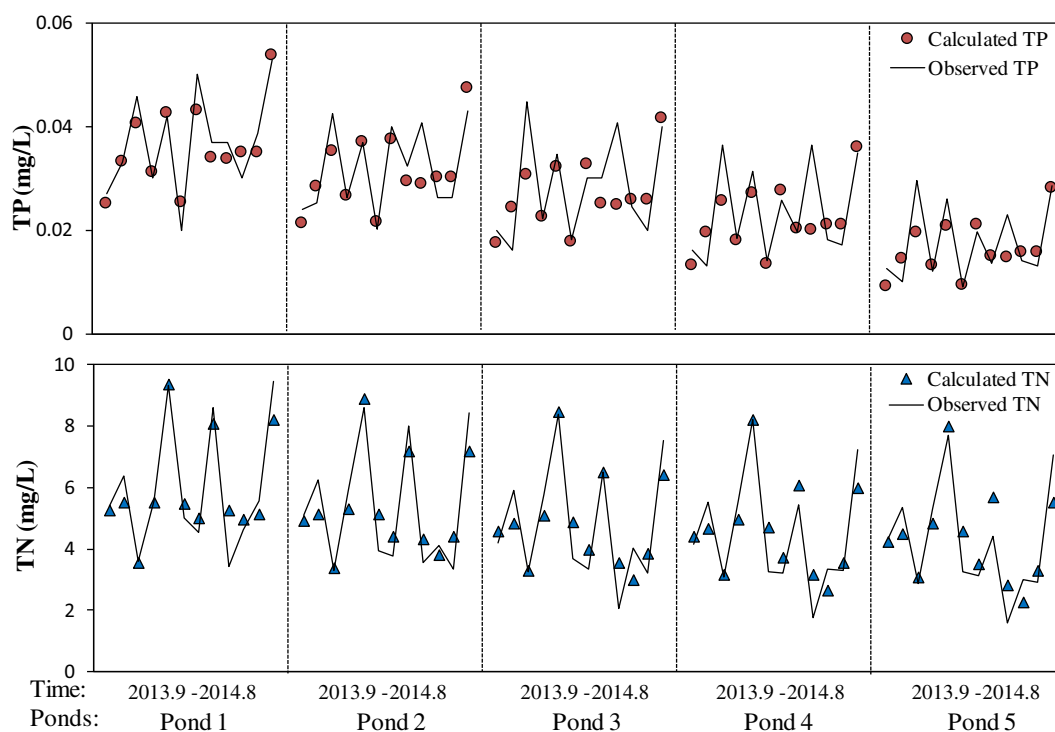


Fig. 2. Monthly time series of observed and calculated TP and TN in original MPS.

denitrification and plant uptake and to ultimately enhance the nitrogen and phosphorus removal of the MPS.

3.2. Effects of individual EFBs and FBs on nutrients removal

In the enhanced MPS, P and N removal was enhanced by the different units of EFBs and FBs such as the plants and the different carriers. To the further study these different units of EFBs and FBs, laboratory batch experiments including plant experiments of *Myriophyllum verticillatum*, denitrification experiment and adsorption experiments were used to calculate the parameters of the units.

3.2.1. Denitrification effect

The denitrification curves of the three carriers in different seasons based on the denitrification batch experiments are presented in Figs. 3 and S2. When exposed to zeolites, the concentration of TN decreased gradually over time. The time taken to fall to a constant concentration varied through the different seasons. Similar trends were found in the ceramsite and the stereo-elastic packing. In addition, the denitrification rates of the different carriers in different seasons were calculated (Table 1). It is evident that the denitrification rate was highest in summer and lowest in winter, indicating that the denitrification rate increased with temperature, which is consistent with the findings of other studies (Capua et al., 2017). Denitrification is a biological process and the optimal temperature for denitrifiers ranged from 30 to 40 °C (Zheng et al., 2012). Compared with the denitrification rates of carriers in other studies at high temperatures such as 0.058 for granules (Table 2), the denitrification rates of carriers in this study were found to be 0.069–3.99, demonstrating the better microbial activity of denitrification bacteria in summer. The advantages of the carriers compared with other carriers (Table 2) at about 20 °C also indicate that the process of cultivating biofilm using carriers in this study was successful.

Moreover, the effects of temperature differed with each carrier. Although the denitrification rates of the stereo-elastic packing were similar in the spring, autumn and winter, the denitrification rates of the zeolites and ceramsite were significantly higher in the spring and autumn than in the winter. In general, denitrification rates were low at

temperatures below 20 °C (Yi et al., 2009b). The porous carriers may be more conducive to denitrification at a lower temperature than stereo-elastic packing, possibly because the robust denitrifying bacterial consortium formed by the porous structure reduced the negative impact of low temperature.

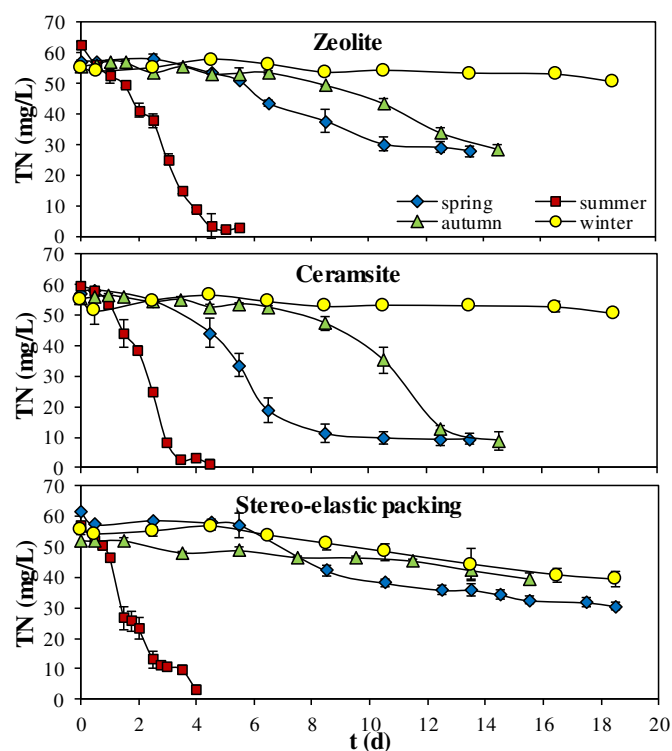


Fig. 3. Variations in concentrations of TN of different season in denitrification experiment by various carriers.

Table 1
The denitrification rate of various carriers in different season.

mgN/g-carrier/d	Ceramsite	Zeolite	Stereo-elastic packing
V _D in spring (18 °C ^a)	0.059	0.016	0.88
V _D in summer (30 °C ^a)	0.16	0.069	3.99
V _D in autumn (18 °C ^a)	0.045	0.014	0.52
V _D in winter (5 °C ^a)	0.010	0.0045	0.46
θ	1.095	1.095	1.075
V _{D20}	0.051	0.018	1.08
k _{d20} ^b	9.21	10.73	6.50

^a The denitrification experiments was carried at this temperature.

^b The unit of k_{d20} is mgN/m²/d.

Compared with the θ values of 1.14 observed in floating treatment wetlands and θ values ranging from 1.05 to 1.18 in CWs (Beutel et al., 2009; Gao et al., 2017), the θ values for the nitrogen removal of the carriers were 1.075–1.095 in this study. This indicates that the carriers in the EFBs and the FBs were less sensitive to temperature because of the comparatively dense distribution of carriers compared with other floating treatment wetlands and CWs.

The denitrification kinetics of the different carriers in different seasons were verified by zero- and first-order kinetic models (Table 3). The zero-order reaction better described the denitrification process (R² > 0.92) than the first-order reaction which had lower R² values of 0.863. The results of this study are similar to those of previous studies (Angar et al., 2016). In other words, in the tank-in-series model of the enhanced MPS, the denitrification rates of the EFBs and FBs were calculated based on zero-order kinetic models. Using the k_{d20} values of the three carriers (Table 1) and the area of each of the EFBs and FBs, the k_{d-bed} values of the EFBs and FBs in every pond were calculated.

3.2.2. Adsorption effect

Fig. S3 presents the adsorption effect of phosphorus and nitrogen by zeolite at different temperatures as determined by adsorption experiments. It is clear that temperature had little effect on the adsorption of phosphorus and nitrogen, especially at low initial concentrations. The effect of temperature can therefore be ignored in practice and the parameters were set at 30 °C. This demonstrates that the retention time of FBs is sufficient to achieve adsorption equilibrium.

A coefficient was introduced to express the difference between the zeolite particle size in the experiments and in practice. The coefficient was estimated to be 0.1 according to the results of many previous studies of zeolite particle size (Cyrus and Reddy, 2011; Piirtola et al., 1998). Because of the different areas of FBs in different ponds, the adsorption effect can be rewritten as follows according to the coefficient and Eqs. (2)–(3):

$$f(\text{CO})_{\text{P-adsorption}} : C_0 = C_e + 0.0855 \frac{V_i}{V} C_e^{1.072} \quad (35)$$

$$f(\text{CO})_{\text{N-adsorption}} : C_0 = C_e + 0.2242 \frac{V_i}{V} C_e^{1.599} \quad (36)$$

Table 2
Summary of the denitrification rate of various carriers in other studies.

Support media	Media sources	Temperature (°C)	Denitrification rate (mg/g/d)	Reference
Periphytic biofilms	A eutrophicated lake	15–35	4.8–12	(Zhao et al., 2018)
Sandy loam	Rainfall	22	0.029	(Sextstone et al., 1985)
Clay loam	Rainfall	22	0.035	(Sextstone et al., 1985)
Sand	Synthetic wastewater	20	0.009–0.012	(Xu et al., 2013)
Granule	Synthetic wastewater	28	0.058	(Zhong et al., 2014)
Sediment	Reservoir	22–25	0.0012–0.023	(Huang et al., 2013)
Ceramsite	Pond	20	0.051	This study
Zeolite	Pond	20	0.018	This study
Stereo-elastic packing	Pond	20	1.08	This study

where V_i is the volume of zeolite in Pond i, V is the total volume of zeolite in all ponds.

The removal quantities by adsorption can be calculated according to Eqs. (35)–(36) and the inflow concentration of FBs. However, because the zeolite was evaluated in the final stage of the enhanced MPS, the adsorption capacity was underestimated.

3.3. Superposition effect of the EFBs and FBs in nutrients removal

Considering the effect of the original MPS and the EFB and FB enhancements in the enhanced MPS, the average observed TP and TN concentrations of the outflow determined by the field experiments were 0.011 and 2.38 mg/L and those of the inflow were 0.037 and 5.04 mg/L, respectively (Fig. S1). Although the TN concentration of the outflow in the enhanced MPS did not reach the prescribed target (Class V surface water quality standard), the removal rates of TP and TN increased from 59.2% and 37.1% to 71.4% and 51.0%, respectively, indicating that the EFBs and FBs were beneficial to enhancing nutrient removal in the MPS. However, the contribution of each EFB and FB cannot be clearly determined from only the enhancement of the removal rate. Moreover, the removal rate of the enhanced MPS was underestimated when the concentration of the inflow was lower than that in the original MPS.

According to the tank-in-series model, the average calculated TP and TN concentrations were 0.012 and 2.48 mg/L and the average calculated TP and TN removal rates were found to be 73.0% and 49.4%, respectively, which were approximately equal to the observed values of the field experiments (Fig. 4). Moreover, the NSE values for the TP and TN in the enhanced MPS were 0.78 and 0.83 respectively, indicating that a good fit was observed between the observed and calculated values and that the parameters were capable of simulating the nutrient removal behavior of the enhanced MPS.

As determined by Eqs. (22)–(25), in one year, the contribution of EFBs to TP removal in Ponds 1–5 is 0.26%, 1.91%, 1.2%, 1.13% and 2.33%, while that to TN removal is 0.059%, 6.22%, 2.79%, 2.29% and 4.94%, respectively. Similarly, in one year, the contribution of FBs to TP removal between Ponds 1–5 is 2.95%, 2.21%, 1.89% and 1.97%, while that to TN removal is 4.32%, 2.91%, 3.08% and 3.40%, respectively (Table 4), indicating that the TP and TN removal rates of the MPS were improved by the superposition effect of the EFBs and FBs. The mechanisms of TP removal in the EFBs and FBs were absorption by plants (43.08%) and adsorption by zeolites (56.92%). TN removal was principally enhanced by denitrification, adsorption by zeolites and plant uptake. Of these mechanisms, the denitrification of the EFBs and FBs accounted for 52.04% and 33.15% of the total TN removal, respectively, and the proportions of adsorption by zeolites and plant uptake were 12.53% and 2.28%, respectively.

In general, the removal of TN by EFBs depends on the plants species, absorption and the microorganisms that attach to the roots of the plants and the substrate (Wang et al., 2009; Wu et al., 2016). In this study, as bio-carriers, ceramsites and stereo-elastic packing were also added into the EFBs for to enhance the performance of N removal and were the main sites for microbial enrichment. Ceramsites are porous carrier

Table 3
Denitrification characteristic of TN removal for different carriers.

		Zeolite				Ceramsite				Stereo-elastic packing			
		Spr.	Sum.	Aut.	Win.	Spr.	Sum.	Aut.	Win.	Spr.	Sum.	Aut.	Win.
Zero-order	K_0	0.105	0.568	0.103	0.018	0.267	0.733	0.23	0.013	0.086	0.735	0.03	0.036
	R^2	0.940	0.974	0.938	0.962	0.928	0.949	0.941	0.962	0.968	0.959	0.944	0.941
First-order	K	0.003	0.02	0.003	0.000	0.009	0.034	0.009	0.000	0.002	0.027	0.000	0.001
	R^2	0.933	0.886	0.890	0.962	0.861	0.777	0.889	0.96	0.971	0.958	0.934	0.935

particles that can utilize the anaerobic environment while appearing in the internal region of a bio-carrier under aerobic conditions (Xing et al., 2000). Chen et al. (2014) developed a reactor that used ceramsite as a carrier and a peak denitrification rate of 7.41 mgNO₃-N/L/h was observed at a nitrate loading rate of 130 mgNO₃-N/L/h. Ceramsites contributed to 22.9% of the denitrification of EFBs, while the stereo-elastic packing contributed 77.1%. Stereo-elastic packing is one of the most effective carriers for micro-polluted water treatment (Feng et al., 2015). It has a larger surface area than plant roots, and can sustain the growth of more bacteria (Wu et al., 2016). In previous studies, the nitrogen removal rate of EFBs increased from 35.3% to 49.2% with the addition of a hanging filler (Wu et al., 2016), and Liang et al. (2013) reported that the biofilms adhering to elastic fillers during the process of autotrophic denitrification were dense and integrated and that anaerobic biofilms appeared later.

Zeolites have been widely used as effective adsorbents for waste water treatment, such as for the adsorption of PO₄-P and NH₄-N, because of their capability of cation exchange (Feronato et al., 2015). The PO₄-P recovery and NH₄-N exchange capacities of zeolite do not affect each other (Guaya et al., 2015). Although the adsorption of PO₄-P by zeolites accounted for a large proportion of enhanced phosphorus removal, we believe that the adsorption potential of zeolites is limited because of the low PO₄-P concentration in the inflow. Compared with the slight contribution to NH₄-N removal by zeolite adsorption, the denitrification of zeolite is more significant. Zeolites exhibited a high removal

capacity of NO₃-N because they are characterized by large volumes of micropores which makes them ideal for supporting the growth of denitrifying bacteria (Jovanovic et al., 2016). In previous studies, it was found that for water with low NH₄-N concentration and high NO₃-N concentrations, compared with the limited adsorption ability of NH₄-N, zeolites exhibited a high removal capacity of NO₃-N (Chen et al., 2013).

4. Conclusion

This study demonstrated the excellent effect of EFBs and FBs integrated into the existing MPS for enhancing TN and TP removal to improve the outflow water quality. For each EFB or FB unit, its function might be little but when a series of them were installed in the MPS, their superposition effect became sufficiently great, as shown by the calculations using a tank-in-series model. The EFBs totally contributed to 23.3% and 8.12% of increases in TN and TP removals, respectively, mainly due to denitrification and plants uptakes, while the FBs totally contributed to 19.6% and 10.7% of increases in TN and TP removals, respectively, mainly due to denitrification and adsorption. The selection of packing materials with large specific surface area for microbial growth and sufficient capacity for adsorption was the key point for significantly improving the MPS's function without large scale engineering measures but only utilization of the space within and in-between the existing ponds.

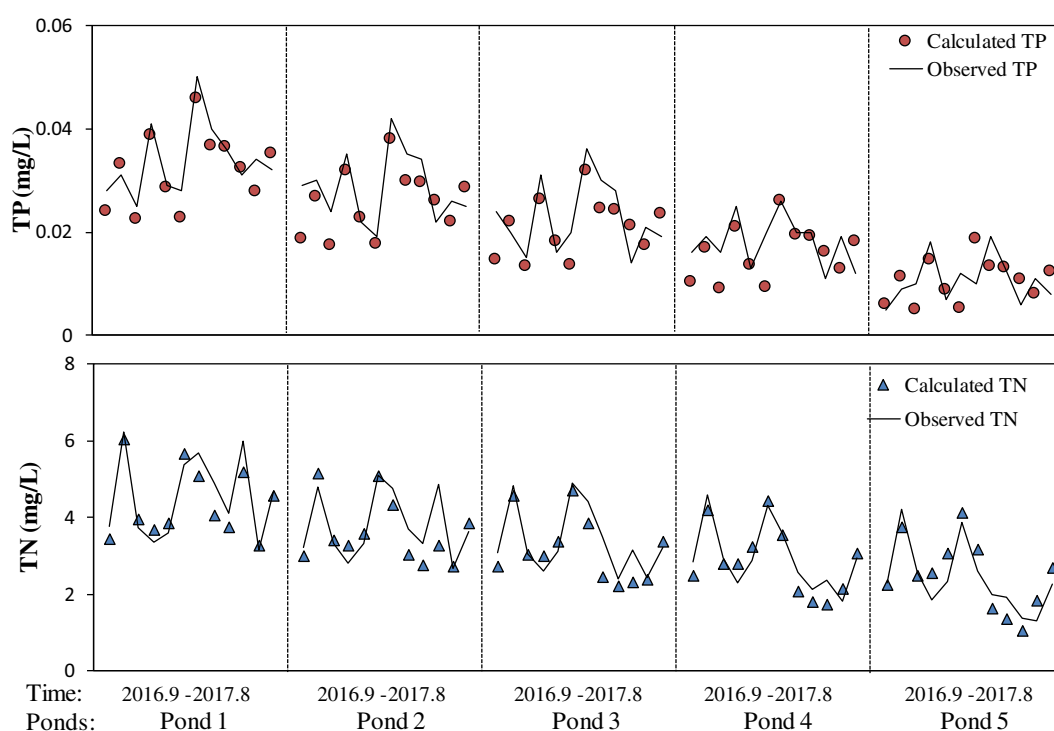


Fig. 4. Monthly time series of observed and calculated TP and TN in enhanced MPS.

Table 4

The contribution of each unit in TP and TN removal in enhanced MPS.

	%	TP ₁	Total	TN ₁	TN ₂	Total
Pond 1	EFB1	0.081		0.0081	0.0084	
	EFB2	0.048	0.26	0.0049	0.0030	0.059
	EFB3	0.13		0.013	0.022	
	FB1	2.95	2.95	1.06	3.25	4.31
Pond 2	EFB1	0.40		0.043	1.13	
	EFB2	0.72	1.91	0.072	3.15	6.22
	EFB3	0.32		0.032	0.61	
	EFB4	0.47		0.043	1.13	
Pond 3	FB2	2.21	2.21	0.90	2.02	2.91
	EFB1	0.56	1.20	0.056	1.15	2.79
	EFB2	0.64		0.064	1.51	
	FB3	1.89	1.89	0.85	2.23	3.08
Pond 4	EFB1	0.52	1.13	0.053	0.93	2.29
	EFB2	0.60		0.061	1.24	
	FB4	1.97	1.97	0.94	2.45	3.40
	EFB1	0.48		0.048	0.79	
Pond 5	EFB2	0.60	2.33	0.060	1.23	4.94
	EFB3	0.68		0.069	1.59	
	EFB4	0.56		0.056	1.08	

TP₁, TN₁: The phosphorus and nitrogen removal by plant absorption or zeolite adsorption.
 TN₂: The nitrogen removal by denitrification.

Declaration of Competing Interest

We declare that we do not have any commercial or associative interest that represents a conflict of interest in connection with the work submitted.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.scitotenv.2019.133678>.

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