



Phosphorus removal enhancement of magnesium modified constructed wetland microcosm and its mechanism study



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ABSTRACT

Over the past few decades, low phosphorus removal efficiency hampered the further application of constructed wetlands (CWs). In this study, magnesium (Mg)-containing materials, i.e., magnesia and magnesite, were added into CWs substrate to improve TP removal performance. Results showed that the average TP removal efficiency of magnesia group (MA-CW) reached 93.3%, which was 17.5% and 18.6% higher than that of magnesite group (ME-CW) and control group (QZ-CW), respectively. P fractionation analysis showed that the proportion of phosphorus sequestration by substrate in MA-CW was 90.4%, while it was only 65.1% and 58.8% in ME-CW and QZ-CW, respectively. Phosphorus assimilation by plant in ME-CW accounted for 9.52%, higher than other groups, which was attributed to the stimulated plant growth caused by Mg addition. The average total chlorophyll content of plants in ME-CW was $4.61 \text{ mg g}^{-1} \text{ FW}$ during the study period and the plant growth rate was superior to other groups, whereas in MA-CW, excessive alkaline ($\text{pH} = 10.29$) caused by MgO dissolution depressed plant growth. Mg addition also elevated the absolute abundance of (PAOs), which followed the order of MA-CW ($1.76 \pm 0.03 \times 10^7 \text{ copies g}^{-1}$) > ME-CW ($1.35 \pm 0.02 \times 10^7 \text{ copies g}^{-1}$) > QZ-CW ($4.48 \pm 0.13 \times 10^6 \text{ copies g}^{-1}$). Characterization of magnesia and magnesite before and after phosphate adsorption was compared and the results indicated that phosphorus adsorption by magnesium mainly relied on coprecipitation of $\text{Mg}_3(\text{PO}_4)_2$ and MgHPO_4 , as well as ligand-exchange on the surface.

1. Introduction

In recent years, excessive discharge of phosphorus into aquatic ecosystem led to serious eutrophication, which further induce degradation of water quality [1]. Conventional wastewater treatment technologies were insufficient to meet the more and more stringent environmental regulations for phosphorus discharge, which required phosphorus concentration in treated sewage effluents as low as 0.05 mg L^{-1} [2]. High phosphorus concentration was still being detected in rivers and coastal waters into which the secondary effluents from wastewater treatment plant were poured. Consequently, more attention should be drawn to advanced treatment processes to refine treated sewage effluents and narrow its distance with the discharge standards, and eventually achieve compliance.

As a promising technology, constructed wetlands (CWs) have been regarded as a prime candidate for water purification from treated

sewage effluents due to its advantages of environmentally sustainable, simple operation and cost affordable [3]. As summarized by C. Vohla et al. [4], phosphorus removal efficiency in CWs were in ranges of 32.0%–78.4%. The relative low phosphorus removal efficiency of CWs could not alleviate water eutrophication and further limited its widespread application. Thus, intensify phosphorus removal in CWs is of great importance.

The phosphorus removal mechanisms of CWs mainly include substrates adsorption, plant uptake, as well as stabilization by microbial growth [4,5]. As reported in previous studies, phosphorus removal through substrate accumulation accounted for 36.2–87.5% of phosphorus input, while plant and microbial uptake accounted for about 1.3–26.4% [6,7]. Nevertheless, common substrate (e.g. gravel, zeolites and ceramsite) usually showed low phosphorus adsorption ability, which largely limited phosphorus removal efficiency of CWs. A considerable deal of interest has been paid to modified wetland substrates

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containing calcium, aluminum and iron to enhance phosphorus removal performance in CWs. Accordingly, fragmented Moleanos limestone, mainly composed by CaO (55.6–55.8%), showed superior phosphorus removal efficiency due to the stable chemical precipitation of hydroxyapatite [8]. Another study of C. Barca et al. [9] showed that steel slag filter removed over 88% of the influent total phosphorus (TP) with the main mechanism of CaO dissolution, followed by the precipitation of tricalcium phosphate, octacalcium phosphate and hydroxyapatite. Beside, aluminum-based water treatment residual also showed good phosphorus removal capacity in engineered wetlands because the presence of amorphous aluminum species [10]. And sponge iron was proposed for phosphorus adsorption from wastewater, due to the significant flocculation effect of Fe(OH)₂ and Fe(OH)₃, which could bind phosphorus easily [11]. In addition, some substrates rich in manganese was recommended which was related to redox processes for phosphate removal [12].

However, to date, few researches applied Mg-containing materials in CWs to upgrade the effectiveness of phosphorus removal. As is well known that Mg is essential to plant photosynthesis, which could transfer oxygen from leaves to roots in aquatic plant [13,14]. In addition, Mg²⁺ participated in the process of phosphate accumulation by polyphosphate-accumulating organisms (PAOs) [15]. Furthermore, there was a potential for struvite (MgNH₄PO₄·6H₂O) formation when concentrations and pH were favorable, which enlightened the purpose of recovering phosphorus and nitrogen simultaneously [16]. Although previous research indicated that magnesium oxide might had a good potential for phosphorus adsorption, presently there is no comprehensive study on intensified TP removal performance in CWs by the addition of Mg-containing materials.

In general, the objectives of this study were (1) to investigate the performance of phosphorus removal by magnesium modified CWs; (2) to illuminate the mechanism of phosphorus removal in magnesium modified CWs; (3) to examine the influence of Mg on plants growth and microbial communities in the system.

2. Materials and methods

2.1. Physical and chemical properties

Two different Mg-containing substrates, i.e., natural magnesite and sintered magnesite, were investigated in the present study. Natural magnesite, exploited from Haicheng of Liaoning province in China, was rich in magnesium carbonate (MgCO₃), while sintered magnesite, manufactured by natural mineral magnesite through high-temperature calcination, was mainly composed of magnesium oxide (MgO). The main chemical composition of them are shown in Table 1. All Mg-containing substrates were air-dried and grounded to pass through 3-mm mesh sieve.

Table 1
Chemical composition of magnesite and magnesite substrate samples (wt.%).

Components	Silica sand	Sintered magnesite	Natural magnesite
MgO	< 0.06	86.15	N.D.
MgCO ₃	N.D.	N.D.	46.32
SiO ₂	99.39	3.72	1.20
CaO	0.07	1.37	0.84
Fe ₂ O ₃	0.06	0.69	0.28
Al ₂ O ₃	0.25	0.28	0.16
Na ₂ O, K ₂ O, TiO ₂	0.17	N.D.	N.D.
LOI ^a	–	7.79	51.20

N.D. = no detect.

^a Loss on ignition.

2.2. Constructed wetlands design and operation

Three CW microcosms, constructed by polyethylene barrels (35 cm in diameter and 40 cm in depth) was operated side by side from 8th June to 10th September in 2016. The construction of microcosms was rain protected, but still exposed to natural variations in air temperature and light exposure. Briefly, three groups: control group (QZ-CW), magnesite group (MA-CW) and magnesite group (ME-CW) were established. These microcosms all chose gravel (3–4 cm particle size) as a 5 cm bottom layer; two contained a 30–70% mixture of silica sand with magnesite and magnesite (2–3 mm particle size) respectively, reaching an effective depth of 30 cm, while in another microcosm the magnesite or magnesite was completely replaced by silica sand as a control. The physical properties of these substrates such as porosity and bulk density were determined by standard methods [10,17]. As for the chemical properties of each substrate, pH was measured by a pH meter (PHS-3D, Rex Instrument, Shanghai, China) followed mixing 2 g of milled material with 20 ml of 0.01 M CaCl₂ and electrical conductivity (EC) was determined using a Radiometer electrical conductivity meter (DDBJ-350, Rex Instrument, Shanghai, China) at 25 °C after mixing 2 g of milled material with 10 ml of deionized water [10,18]. Specifics are shown in Table 2. For each microcosm, young *Phragmites australis* were employed in CWs with the planting density of 124 plant stems m⁻² corresponding to 12 plants in each barrel. A porous PVC pipe was vertically inserted into the middle of substrates to measure specific physical and chemical parameters of wastewater *in situ*, such as dissolved oxygen (DO), air temperature and pH. Simulated sewage effluent wastewater, contained 60 mg L⁻¹ chemical oxygen demand (COD), 8 mg L⁻¹ ammonium nitrogen (NH₄-N), 12 mg L⁻¹ nitrate nitrogen (NO₃-N) and 1.5 mg L⁻¹ total phosphorus (TP), was prepared with tap water. For the trace elements, modified Hoagland nutrient solution without N, P and Mg was selected for growth and cultivation of vegetation [19]. The wastewater with the volume of 8 L was fed onto substrate surface and drained away by gravity through an open valve at the bottom of each wetland every three days, and the hydraulic loading rate (HLR) was 30 L⁻¹ m⁻² d⁻¹.

2.3. Physicochemical parameters determination

Water temperature and DO were determined by DO meter (HQ30d, HACH, USA). pH was monitored using a portable pH meter (PHS-3D, Rex Instrument, Shanghai, China). The water samples from influent and effluent of each microcosm were collected every three days. The samples were filtered through 0.45 μm membrane filters prior to the following analyses. The concentrations of NH₄-N, NO₃-N, NO₂-N and TP were measured using spectrophotometer according to standard colorimetric methods [20].

Substrate samples were collected at three substrate layers: 0–10 cm, 10–20 cm and 20–30 cm at the end of experiment, then mixed homogeneously [6]. Phosphorus content in substrates were measured by molybdenum blue method for substrate solution following nitric acid and sulfuric acid digestion [21]. An X-ray powder diffractometer (D8 Advance, Bruker, Germany) using Cu-Kα radiation was applied to identify the mineralogical composition of solid samples in MA-CW and ME-CW before and after the experiment. The powder samples were scanned from 10 ° to 80 °. The XRD patterns were analyzed by the

Table 2
Physicochemical properties of substrates in CWs.

Items	QZ-CW	MA-CW	ME-CW
Porosity (%)	41.60	44.70	46.70
Bulk density (g cm ⁻³)	1.23	1.29	1.38
EC (μS m ⁻¹)	25.10	340	175
pH	7.22	9.63	8.17

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