Sustainable modulation of anaerobic malodorous black water: The interactive effect of oxygen-loaded porous material and submerged macrophyte

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ABSTRACT

Depleted oxygen (O_2) in the sediment and overlying water of malodorous black water poses a potential threat to aquatic ecosystems. This study presents a method for sustainable regulation of the dissolved oxygen (DO) levels towards the malodorous black water. Oxygen-loaded natural porous materials were prepared by vacuum degassing to remove air from the pores and fill them with pure O_2. Capping anaerobic sediment with the prepared 6 oxygen-loaded porous materials was effective in prompting the DO concentration of the malodorous black water. Although granules activated carbon (GAC) displayed the highest oxygen-loading capability, oxygen-loaded volcanic stone additive was more efficient for long-lasting combating of the anaerobic condition because the DO level at sediment-water interface (SWI) and the DO penetration depth showed approximately 5.38- and 3.75-fold increase, respectively, compared with the untreated systems. The improvement in DO was substantially enhanced in the presence of submerged macrophyte (*Vallisneria natans*), during which the release of O_2 from oxygen-loaded volcanic stone facilitated the plant growth. With the joint efforts of the O_2 released from volcanic stone and photosynthesis by the macrophytes, the DO levels were maintained at approximately 6.80 mg/L after a 41-day incubation, which exceeded (P < 0.05) the value in only oxygen-loaded volcanic stone or macrophytes added treatments. In addition to the elevated DO level, the combined employment of oxygen-loaded volcanic stone and macrophytes triggered a negative ammonia (NH_4^+-N) flux across the SWI and an 85.82% reduction of methane (CH_4) production compared with those without treatment, accompanied by a decrease in total inorganic carbon and a 2.55- fold increasing of submerged macrophyte biomass, which is presumably attributed to nitrification, remineralization, and assimilation. The results obtained here shed a degree of light on the sustainable modulation of the anaerobic condition in malodorous black water.

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1. Introduction

The occurrence of malodorous black water is a phenomenon in which water turns black and produces offensive odorous gas (Talaiekhozani et al., 2016). Due to the extensive input of industrial and municipal waste and runoff in urban and agricultural areas into rivers and lakes, malodorous black water is frequently reported in China, which is a serious natural ecosystem disaster that threatens numerous lakes and rivers nationwide. Currently, the number of bodies of malodorous black waters in China is 2100. As a result, the need to turn back the black tide is vital.

China has made considerable efforts to mitigate the pollution of malodorous black waters. Currently, the constructions of waste-water treatment plants have paid special attention to the disposal of large volumes of wastewater before there are received by lakes and rivers. However, the treatment of malodorous black water is currently not satisfactory due to the high concentration of pollutants present in the discharged water, such as organic matter and nitrogen, even if the required removal efficiency is attained (Jin et al., 2018), which can also enhance pollutant inputs for

malodorous black waters. Moreover, despite the implementation of pollution abatement and control programs, the contaminants in the water column will mitigate to the bottom and sequentially gather in the sediment in the polluted waters; thus, the sediment become a pollution source in the aquatic systems (Borgnino et al., 2006; Ding et al., 2015, 2018). Without the application of post-treatments to remove the pollutants in the sediment and implement ecological restoration, the tide of the malodorous black water can hardly be reverted.

Techniques using biotechnological and engineering methods to remediate malodorous black water have been received considerable attentions. Removal of surface sediment has been considered as an alternative to reduce nutrients release from anaerobic sediments (Lürling and Faassen, 2012; Waajen et al., 2016). Ex-situ dredging of the anaerobic sediments is extensively employed for sediment remediation. Despite the encouraging results of some laboratory studies, some field experiments have reported a minimal reduction in sediment nutrients flux after dredging in the long term (Kleeberg and Kohl, 1999; Oldenborg and Steinman, 2019). Recent studies evaluating the effect of sediment dredging on water quality improvement after dredging for 6 years discovered that the decomposed algae can precipitate and settle on the lake sediment in summer and autumn, which favored the release of phosphorus from sediments by the exhaustion of oxygen, reduction of Fe oxyhydroxides to Fe(II) (Han et al., 2015), and releasing of algaloriginated phosphorus (Ai et al., 2019), hence impaired dredging effectiveness (Chen et al., 2018).

Malodorous black water is also characterized by hypoxic (low dissolved oxygen (DO) concentration) and high content of organic matter and ammonia (NH_4–N) (Zhou et al., 2015), which in turn is a key factor that affects the degradation of organic matter and the biogeochemical cycle of nitrogen (Svensson et al., 2015). A conventional approach to increasing oxygen (O_2) levels in waters is mechanical aeration, which is capable of reducing organic matter since it will cause the proliferation of organic degraded-related bacteria (Yuan et al., 2018). However, this methodology is becoming obsolete due to the high energy consumption and relatively low efficiency (Podsiadłowski et al., 2018). Also, aeration can substantially enhance the re-suspension of the polluted sediment and prompted the light attenuation in the aquatic ecosystem. An alternative approach involves chemicals addition, which may donate oxygen as reacting with water. Hydrogen peroxide (H_2O_2) can decay to water and oxygen via chemical and biological oxidation–reduction reactions (Matthijs et al., 2012), and was assumed to be a potential way to improve the oxygen levels in water, whereas H_2O_2 had to be continually supplied and would kill the degrading microbes at high concentrations (Nykänen et al., 2012). The potential negative impact of H_2O_2 can be mitigated using granulated calcium peroxide (CaO_2, which can release 17% of its weight as O_2) as a slow oxygen releasing compound (Wang et al., 2019b). When the granulated CaO_2 was spread to the lake, the effect was visible for the entire 40-week experiment (Nykänen et al., 2012); however, some attempts to improve the DO concentration using CaO_2 caused an increase in the pH levels from 7.1 to 11.3, due to the generation of $Ca(OH)_2$ (Lee et al., 2014; Zhou et al., 2019).

Considering the energy input, total environmental impact and public acceptance, the exploration of high-efficient, cost-effective, and most importantly, environmentally benign methods is recommended to treat the malodorous black waters, of which the improvement in DO concentration should be given high priority. As previously addressed, the level of DO is essential for the survival of aerobic microbe and aquatic species, including submerged macrophytes. In desired aquatic systems, particularly those dominated by submerged macrophytes, the deficiency of some

nutrients and high DO concentration may occur due to the assimilation of nutrients and photosynthesis by the macrophytes. Once established, submerged macrophytes are considered to have a positive effect on water clarity and the stability of aquatic ecosystems by a variety of buffer mechanisms (Stephen et al., 1998). Although the recovery of aquatic vegetation was determined to be crucial for restoring malodorous black water, the oxygen exhaustion or at the minimum level (<0.2 mg/L) (Zhu et al., 2017), and the extreme high content of organic matter, ammonium and alkaline condition (Wang et al., 2017b), deteriorated the growth of aquatic vegetation. From our perspective, emerging technologies that may trigger the recovery of the self-organized ecosystems once the implementation of pollution abatement was achieved, should be investigated.

The two microcosm experiments presented in this paper are concerned with sustainable improvement of DO level in malodorous black waters. We prepared oxygen-carried porous material that can release oxygen with limited negative environmental impacts. With the modified microenvironment particularly DO levels at the sediment-water interface (SWI) with oxygen-loaded porous material capping, submerged macrophytes were subsequently incubated. This study sought to develop and test a method for increasing the DO level of sediments and regulation of the anaerobic environment with the joint efforts of oxygen-carried porous material and submerged macrophytes, and explore how the aquatic ecosystem responded.

2. Material and methods

2.1. Materials and characterization studies

Granular activated carbon (GAC), akadama clay, pumice stone, ceramsite, volcanic stone and zeolite and were used to load oxygen. The 6 natural porous materials were obtained from Beixing Natural Minerals Ltd., Chongqing, China. The specific surface area of the porous materials was determined by the Brunauer−Emmett−Teller (BET) method with a Micromeritics ASAP-2020 apparatus (Micromeritics Inc., USA). The pore volumes were obtained by analyzing the 77 K/N_2 adsorption−desorption isotherms, during which the analysis was performed at a relative pressure (P/P_0) of 0.98. The pore size distribution of the materials were calculated from the N_2 adsorption−desorption isotherms using the non-local density functional theory (NLDFT) model for carbonaceous slit-like pores implemented in Belsorp software (Heo and Park, 2018).

2.2. Preparation of oxygen-carried porous materials

The porous materials were washed with double deionized water and dried at 90 °C. The materials were degassed in a sealed container for 5 min at room temperature under vacuum (−0.09 MPa) to remove air from their pores. Then pure O_2 was subsequently pumped into the container and maintained at a pressure of 0.2 MPa for 5 min to enable the O_2 to enter the pores.

Oxygen-loading capability of the porous materials was acquired from the O_2 adsorption−desorption isotherms which recorded at 77 K using a Quantachrome analyzer. The porous materials were dried at 200 °C for 5 h before measurement. Oxygen uptake under dynamic pressure (from 4 to 74 bars) was performed by the H-sorb 2600 high pressure volumetric adsorption analyzer (Gold APP Instrument Corporation, Beijing, China). A known amount of oxygen was dosed into the measurement tube that contained the porous materials. When equilibrium between the materials and adsorbed oxygen is attained, a comprehensive final equilibrium pressure can

be automatically recorded. The quantity of adsorbed O_2 was calculated from the generated data (Ahmed et al., 2017).

Then two microcosm experiments were established to assess the DO dynamics and evaluate the joint effects of the oxygen-loaded porous materials and submerged macrophyte on sustainable DO improvement.

2.3. Microcosm experiment 1: evaluation of DO improvement of the oxygen-carried porous materials

A microcosm scale study was established to assess the DO dynamics as function of oxygen-carried porous materials additions. Fourteen Plexiglas cylinders with an inner diameter of 10 cm and height of 60 cm were filled with the same amount of sediment (thickness of 10 cm) and water (3.5 L, a depth of 45 cm), which was collected from a malodorous black lake (29°36′52″ N, 106°19′40″ E) located in Shapingba District, Chongqing, China, to simulate the environmental malodorous black scenario. The chemical characteristics of the water are DO 0.20 mg/L, NH_4^+-N 9.01 mg/L, total organic carbon (TOC) 20.08 mg/L, total inorganic carbon (TIC) 25.07 mg/L and pH 7.24 (assessed using PHS-3C, China). The 14 columns were randomly divided into 7 treatments: two columns that contain water and sediment were used as controls, and then sediment in other columns was capped by oxygen-loaded GAC, akadama clay, pumice stone, ceramsite, volcanic stone and zeolite, respectively, with a dose of 0.8 kg/m^2 and with duplicates for each. A DO meter (Yellow Springs Instruments (YSI, Proplus)) was lightly placed into the overlying water and held at 1–2 cm above the sediment surface to assessed the DO level in each column. During the measurements, the O_2 probe was gently circulated (Bonaglia et al., 2019) to prevent the electrode from consuming a portion of oxygen at the moment it touches the water, and the measurement was conducted twice a day. Each experimental column was wrapped with black plastic to shield the ambient light below the sediment surface. When the DO concentration in the majority of the columns displayed slight alternations, the DO profiles across the SWI in the control and the column that showed the most satisfactory DO improvement were assessed using an O_2 microelectrode (Unisense A/S, Denmark), which had a tip diameter of 100 μm and was protected by a glass tube with inner diameter of 3 mm. The diameter of the glass tube exceeded the size of the porous materials to avoid damage of the sensor by the materials. Then the microelectrode was moved through the capping layers to some depth in the sediment by an increment of 100 μm, with assistance of a Unisense A/S microelectrode positioning system (Fig. S1). Calibration of the DO sensor was performed before each measurement. Three profiles per microcosm were randomly measured in the sediment samples to assess spatial heterogeneities. The position of the SWI was determined from the break in the O_2 concentration gradient (Miao et al., 2017, 2018).

2.4. Microcosm experiment 2: the combined role of oxygen-carried material and submerged macrophyte in DO improvement

The most efficient oxygen-loaded porous materials can be obtained based on the evaluation of the DO concentration both at the overlying water and the DO profiles across the SWI in microcosm experiment 1. Then the joint effects of the oxygen-loaded porous materials and submerged macrophyte on sustainable DO improvement were assessed. Eight Plexiglas cylinders that contain the same amount of sediment and water were divided into 4 groups: 1) containing water and sediment and used as the control, 2) the incubation of submerged macrophyte (*Vallisneria natans* seedling in the surface sediment, 3) capping the surface sediment with the most satisfactory oxygen-loaded porous material

according to experiment 1, and 4) after capping the sediment with oxygen-loaded porous material, the *Vallisneria natans* seedling was incubated. The initial fresh biomass of the *Vallisneria natans* seedling in groups 2 and 4 were 2.20 g, with a height of 4.5 cm (containing root and leaf).

During the incubation, pH and the concentration of DO at the SWI were measured as described in experiment 1. The CH_4 and CO_2 areal fluxes from the 4 treatments were monitored at 5-day interval following using a static chamber method. During gas collection, a plexiglas chamber (with diameter of 14 cm and height of 10 cm) was used to cover the outer of the column and submerge 2 cm into the water contained (Fig. S2), which served to maintain the chamber about 10 cm above the water surface in the column, meanwhile avoided gas exchange between the column and atmosphere. After 3 h, the gas was collected in the gas bag (HB.3-CQD, China) using a pump (WT-80, China) through the sampling hole. Then the CH_4 and CO_2 concentration are assessed by gas chromatography with flame ionization detection (7890B Agilent, USA) (Li et al., 2018) and the CH_4 and CO_2 fluxes are calculated using the method described in our previous literature (Ai et al., 2019).

Ten milliliter of overlying water samples (5 cm above the sediment) were also collected and filtered through a 0.45 μm Sartorius filter prior to analysis of NH_4–N at the beginning and end of the experiment. After each sampling, all the studied systems were gently supplied with the original filtered water to compensate for the water losses due to sampling and evaporation. The NH_4–N fluxes were calculated according to the following mass balance (Zhu et al., 2019):

$$F_n = \frac{\left[VC_a - C_b + \sum_{j=1, i=1}^{n} V_j(C_a - C_i) \right]}{S \times t}$$

where F_n is the flux (mg/m^2 d), V is the volume of overlying water (L), C_a is the NH_4–N concentration (mg/L) in the end, C_b is the NH_4–N concentration at the start, C_i is the NH_4–N concentration of the compensating water (mg/L), V_j is volume of sampling water (L), S is the cross section area of the cylinders (m^2) and t is incubation time (d).

After 41 days of incubation, carbon fractions of overlying water samples were analyzed. Concentrations of total carbon (TC) were determined using the Vario El Cube elemental analyzer (Elementar) equipped with a thermal conductivity detector. The concentrations of TIC were measured with the SoliTIC (Elementar) module coupled with the Vario El Cube. Samples were heated to 50 °C and acidified with 5% HCl. The released CO_2 was measured with a thermal conductivity detector. The TOC was determined by subtracting the TIC from the TC (Wang et al., 2019a; Żarczyński et al., 2019). During the assessment of the fresh weight of the submerged macrophyte at the start and end of the incubation, the submerged macrophyte was cleaned of periphyton under running water and dried on filter paper, followed by immediate measurement of the weight (Jasser, 1995).

The two microcosm experiments were performed at a constant 25 °C in an illumination incubator (GZX-250BSH) with a light intensity of 40 μmol photons/(m^2/s) under 12 h:12 h light and dark cycles.

2.5. Statistical analyses

The Statistical Package of the Social Science 18.0 (SPSS 18.0) was used for statistical analysis. An analysis of variance (ANOVA) was used to test the statistical significance of the results; p values less than 0.05 were considered statistically significant. The conceptual diagram was performed by Adobe Illustrator CS 5.

3. Results

3.1. Characterization of porous materials

Akadama clay and GAC showed an apparent higher surface area (220.89 and 149.17 m²/g, respectively) than other porous materials (Table 1). The total pore volume showed a distinct difference among the investigated materials and followed the order of GAC, akadama clay, volcanic stone, zeolite, pumice stone and ceramsite. In addition to the minimum surface area (0.41 m²/g) and pore volume (0.0014 cm³/g) in ceramsite, the maximum average pore size (12.38 nm) was also observed. The distribution of pore size revealed that the narrow (size less than 2 nm) and the wide pore (greater than 20 nm) accounted for the majority of the pore distribution for GAC. A similar pattern was also seen in pumice stone. Although the surface area (71.22 m²/g) and pore volume (0.11 cm³/g) of volcanic stone were substantially lower than those of akadama clay and GAC, its pore size in the range of 2–20 nm was prevalent. Although the pore size in the range of 2–20 nm was also abundant in zeolite, its low pore volume likely did not benefit the oxygen loading.

3.2. Oxygen-loading capabilities

Under elevated pressure, the O_2 adsorbing capability showed constant increase for GAC, akadama Clay, volcanic stone and zeolite. Conversely, no apparent adsorption by pumice stone and ceramsite occurred, which indicates that O_2 can be reversibly adsorbed in the pores at the chosen pressure. Theoretically, the O_2 adsorbing capability is approximately 0.38 mmoL/g for GAC, which showed the maximum O_2 uptake among the analyzed materials, followed by akadama clay (0.23 mmoL/g), volcanic stone (0.17 mmoL/g), and zeolite (0.16 mmoL/g). Note that O_2 adsorption by volcanic stone maintained an increase with an increase in pressure (Fig. 1).

3.3. Oxygen dynamic in overlying water

Capping sediment with the oxygen-loaded porous materials had a distinct impact on the DO concentrations in overlying water. In the controls, the DO concentration remained at approximately 0.20–1.75 mg/L throughout the 11-days incubation (Fig. 2). Oxygen-loaded zeolite and ceramsite can elevate the DO level to 2.84 mg/L, while the DO concentration showed a dramatic decline in the oxygen-loaded ceramsite capped groups. Although capping sediment with oxygen-loaded zeolite improved the DO level maximum to 3.8 mg/L on day 2.5, the DO showed a constant subsequent decline afterward and an insignificant ($P > 0.05$) difference with the control on day 11. In contrast with the oxygen-loading capability test, oxygen-loaded GAC substantially improved the DO level (6.84 mg/L) when it was added in the systems, while the DO concentration showed a rapid decrease and the final concentration
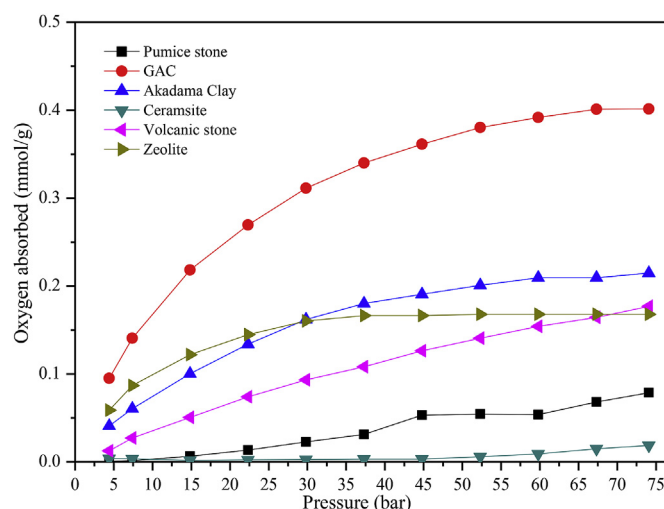

Fig. 1. Oxygen-loading capability of the investigated porous materials. The data are expressed as average of duplicates.
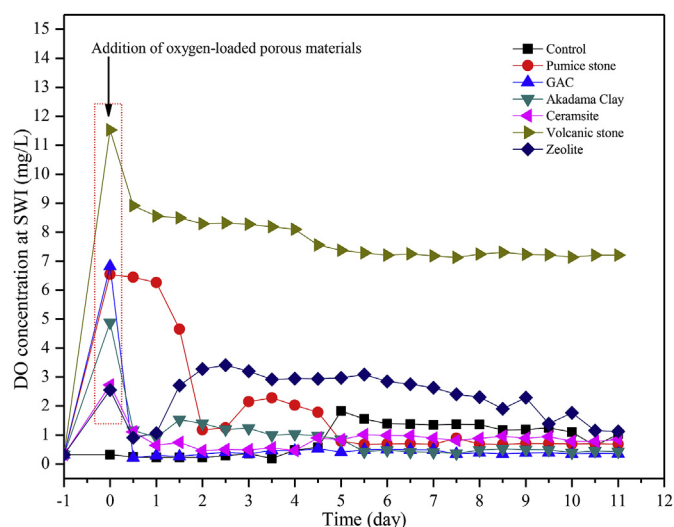

Fig. 2. Dynamic of DO concentrations at the SWI when capping the surface sediment with various oxygen-loaded porous materials during 11-day incubation. The data are expressed as average of duplicates.

was considerable lower than that in the control groups. A similar pattern was also observed in the presence of oxygen-loaded akadama clay and pumice stone. Capping the sediment with oxygen-loaded volcanic stone produced not only maximum DO levels (11.84 mg/L) among the treatments, but also caused sustainable

Table 1
Surface area and porosity parameters of the studied porous materials obtained by the N_2 adsorption.

Materials	S_{BET} (m²/g) [a]	Pore size (nm)[b]	V_{total} (cm³/g) [c]	$V < _{2nm}$(cm³/g)	V_{2-5nm}(cm³/g)	V_{5-10nm}(cm³/g)	$V_{10-20nm}$(cm³/g)	$V_{20-30nm}$(cm³/g)	$V > _{30nm}$(cm³/g)
GAC	149.17	7.52	0.30	0.059	0.047	0.046	0.053	0.0748	0.023
Akadama Clay	220.89	4.80	0.25	0.10	0.070	0.030	0.008	0.013	0.026
Pumice stone	40.34	6.50	0.014	0.0034	0.0050	0.0024	0.00052	0.00075	0.0023
Ceramsite	0.41	12.38	0.0014	0.000081	0.00033	0.00019	0.000097	0.000084	0.00057
Volcanic stone	71.22	5.00	0.11	0.0027	0.04	0.027	0.0043	0.0022	0.0077
Zeolite	15.08	12.14	0.047	0	0.0056	0.0077	0.012	0.016	0.0071

[a] BET surface area.
[b] BJH average pore diameter calculated from desorption data, and.
[c] Pore volume calculated from nitrogen adsorption at $P/P_o = 0.98$.

improvement of the anaerobic sediment during the whole study period, and the DO levels were maintained at nearly 7.00—8.00 mg/L since day 41, which indicated that volcanic stone can slowly release the loaded oxygen and deliver it to the sediment and aqueous phase.

3.4. Oxygen profile across the sediment core

The effect of oxygen-loaded volcanic stone on the improvement of DO concentration was further measured using microelectrodes (Fig. 3). In the bulk aqueous phase of control, DO ranged from 0.22 to 1.64 mg/L during the 11-days experimental period. The oxygen penetration was measured up to a depth of ~2000 μm at the beginning of the incubation (Fig. 3a). Although the DO level at the SWI increased to 1.64 mg/L and an oxygen penetration of 7000 μm as attained after 6 days, the DO level decreased to 1.25 mg/L, with an oxygen penetration depth of 3000 μm, which indicates significant oxygen consumption within the sediment. Capping the sediment with oxygen-loaded volcanic stone substantially reverted the anaerobic tide, and the DO concentration in the bulk aqueous phase was 8.67 mg/L after 6 days of exposure to the capping materials, accompanied by an oxygen penetration depth of 18000 μm. However, after 11 days incubation, both the SWI DO level (reduced to 7.16 mg/L) and the oxygen penetration depth (decreased to 1200 μm) showed a slight reductions compared with day 6 (Fig. 3b). Nevertheless, the results clearly demonstrated that the presence of oxygen-loaded volcanic stone produced a substantially higher oxygen concentration and a deeper O_2 penetration in the sediment.

3.5. Combined efforts of oxygen-loaded porous material and submerged macrophyte on physico-chemical parameters

Fig. 4a illustrates the dynamics of DO concentration against time for four different treatments. The DO concentration constantly remained at approximately 0.21 mg/L during the 41-days study, and the addition of submerged macrophyte did not change the profile due to the decomposition of the plant in extremely anaerobic conditions (Fig. S3). The DO concentrations showed a notable increase once the oxygen-loaded volcanic stone was added; however, it decreased to nearly 2.50 mg/L after 5 days with an insignificant difference between oxygen-loaded volcanic stone capping plus submerged macrophyte incubation treatment. With a continuous release of oxygen bubbles, the oxygen-loaded volcanic stone maximally elevated the DO level to 7.76 mg/L. With the exhaustion of the loaded oxygen, the DO concentration then decreased to 1.85 mg/L after 41 days. Conversely, the joint efforts of oxygen-loaded volcanic stone and submerged macrophyte induced an abrupt increase in DO level, which maximally reached 12.21 mg/L. Despite a reduction to nearly 6.80 mg/L after 21 days, the DO level became stable with time toward the end of the study, which indicates the reverse of the anaerobic condition. Concomitantly, after 41 days of exposure, the NH_4—N flux at the SWI showed a statistically significant different ($p < 0.05$) among the treatments (Fig. 4b). In the combined oxygen-loaded volcanic stone and submerged macrophyte treatment, the NH_4-Nflux from sediment to water across the SWI was negative (-0.013 mg/m^2/d) compared to the continuous release of NH_4—N in other treatments (0.032—0.064 mg/m^2/d).
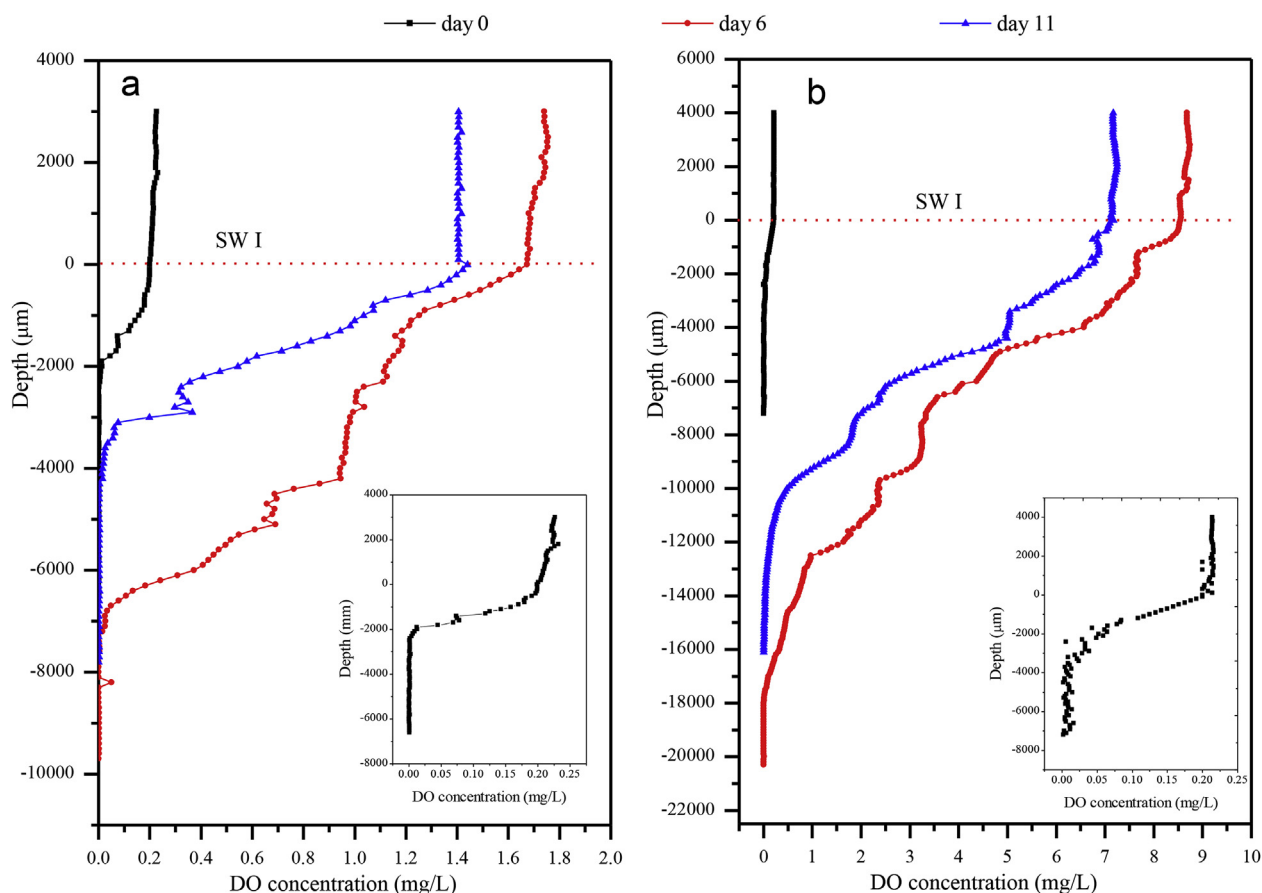

Fig. 3. DO profile across the SWI before and after the addition of oxygen-loaded porous material on the sediment surface. a, control, b, capping the sediment with oxygen-loaded volcanic stone. The data are expressed as average of duplicates. The insert figure illustrated the initial DO profile across sediment core in a narrow range in control and the treatment.
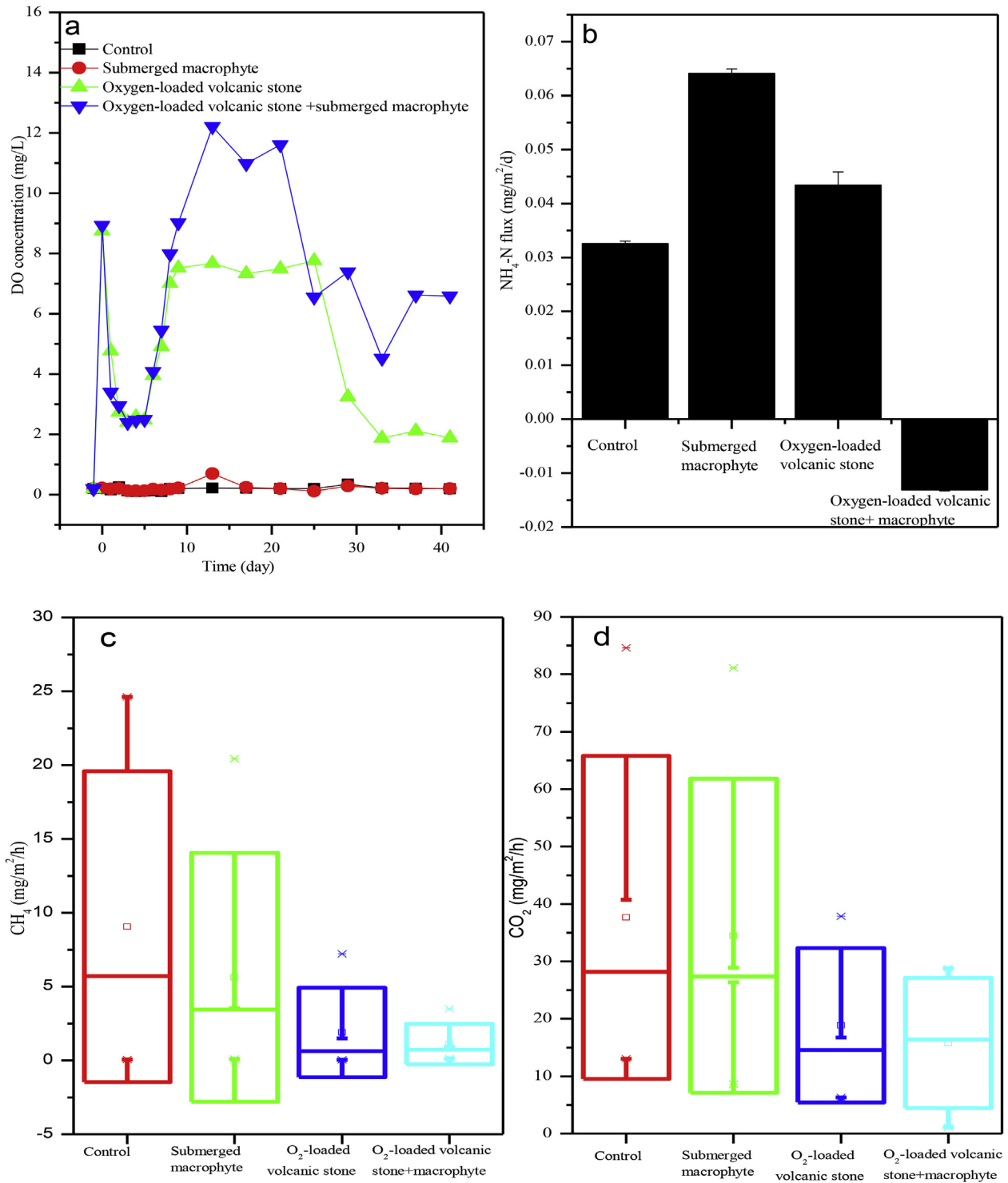
Fig. 4. The dynamic of DO concentration (a), response of NH_4–N flux (b) at SWI, and areal fluxes of CH_4 (c) and CO_2 (d) at the water-atmosphere interface between treatments over the span of 41 days incubation.

The areal fluxes of CH_4 significantly differed among the treatments. In the controls, the CH_4 fluxes ranged from 0.05 to 24.62 mg/m²/h, and the maximum CH_4 fluxes were 1.23, 3.32 and 8.63 times higher than those in submerged macrophyte, oxygen-loaded volcanic stone and oxygen-loaded volcanic stone plus submerged macrophyte treatments, respectively (Fig. 4c). The investigated treatments were effective in reducing CH_4 emission, and the minimum was obtained for the joint efforts of oxygen-

loaded volcanic stone and submerged macrophyte (0.14 mg/m²/h). During the study, CO_2 production was prevalent in the control but showed an abrupt decrease with time. In the oxygen-loaded volcanic stone plus submerged macrophyte treatments, the CO_2 flux showed a pattern that was similar with CH_4 flux, and the released CO_2 was significantly lower than other treatments (Fig. 4d), which suggests that the CO_2 may be restrained in the water column.

Both the TOC concentration and TIC concentration after 41 days incubation were remarkably higher in the controls than other treatments (Fig. 5a). In compared with the control, capping the sediment surface with oxygen-loaded volcanic stone caused a 25 and 52% reduction in TIC and TOC, respectively. The TIC and TOC concentration showed a significant decline ($P < 0.05$) with oxygen-loaded volcanic stone capping; the effects were enhanced with the co-incubation of submerged macrophyte, which implies the remineralization of organic matter in aerobic conditions. In oxygen-loaded volcanic stone capping plus submerged macrophyte added treatments, with an improvement in DO in the systems (also indicated by Fig. 5 d compared with the control, Fig. 5e), organic carbon was converted to inorganic carbon (Fig. 5a), while an abrupt decrease of TIC was observed, indicating the assimilation of carbon by macrophyte. This finding can be confirmed by a 2.55-fold the increase in the macrophyte biomass (Fig. 5b, c, d and f).

4. Discussion

Malodorous black water is generally accompanied by an extremely low DO level and a high NH_4–N concentration. The current main solutions for oxygenation of the hypolimnion and anaerobic sediment include physical methods (e.g., artificial aeration), chemical approaches (e.g., flocculation precipitation), and biological strategies (e.g., ecological floating bed) (Freeman et al., 2018). These approaches may require high cost and energy consumption, and may disturb the settled sediment and induce internal releases of contaminants. Oxygen releasing material, as
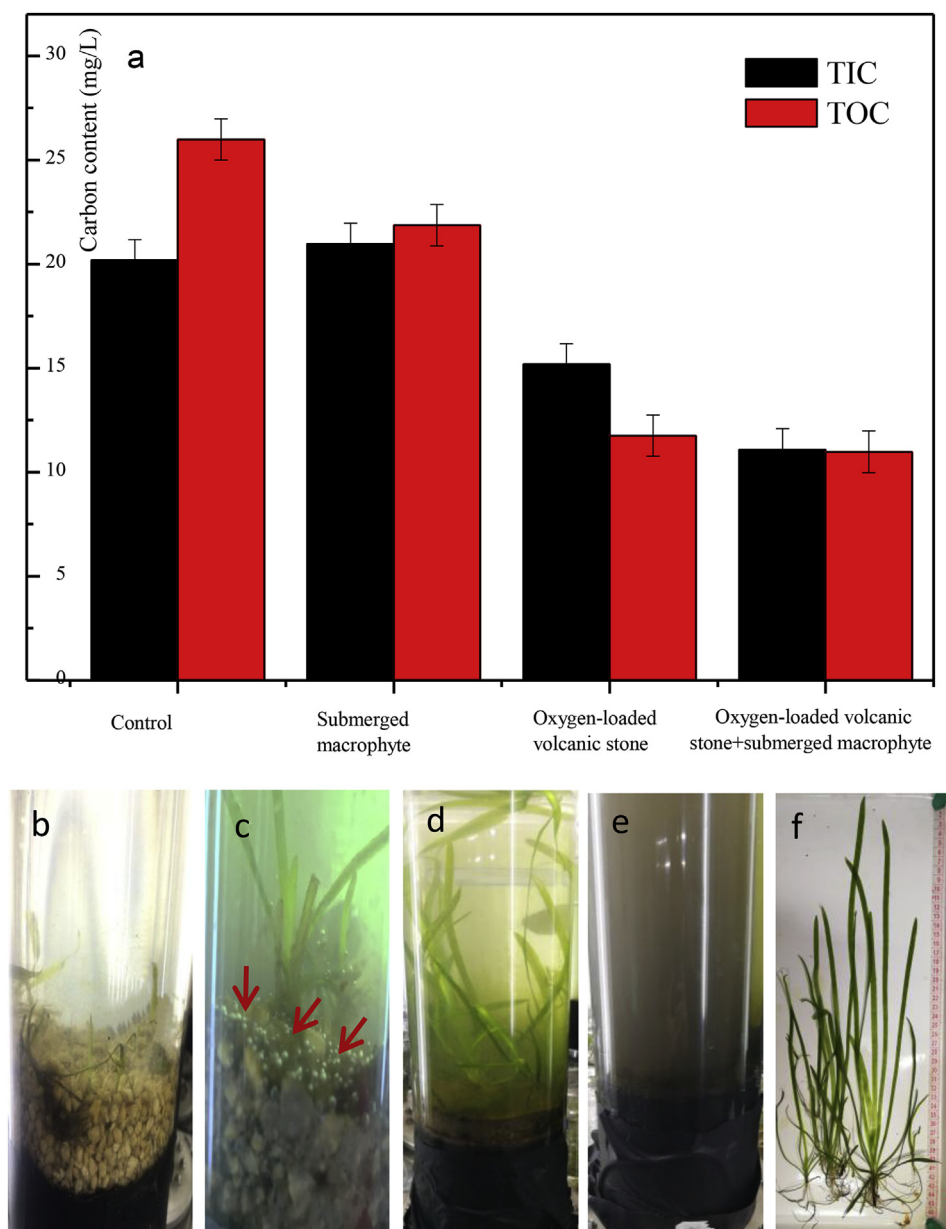

Fig. 5. The TOC and TIC concentration (a) at the overlying water after 41 days incubation. b, c and d describe the growth of *Vallisneria natans* in oxygen-loaded volcanic stone and submerged macrophyte combined treatment at day 0, 6 and 41, respectively. e illustrates the anaerobic condition in control and f displayed the *Vallisneria natans* length after 41 days. The red arrow in c clearly illustrates the formation of oxygen bubble in the capping layer, which favors the growth of submerged macrophyte. d and e also provided visually evidence that the surface sediment showed a yellow color at the end of the study, which was in contrast to a black layer (anoxic) in control. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

represented by CaO_2, has gained great attention due to its ability to generate undesirably high pH in water (Wang et al., 2019b). In this regard, seeking a green, economical, and efficient technology is of great importance (Wang et al., 2019b).,

4.1. Pore-size distribution determines the oxygen-releasing efficiency

In this study, we explored an alternative strategy to deliver O_2 to the anaerobic water that involves the use of porous materials. Natural porous materials are considered to be promising O_2 carriers due to their cost-effectiveness, high surface area and naturally basic surface characteristics (Heo and Park, 2018). We evaluated 6 porous materials for their oxygen loading capability and discovered that GAC possess significantly stronger sorption ability for O_2 (0.38 mmoL/g), followed by akadama clay (0.23 mmoL/g), volcanic stone (0.17 mmoL/g), zeolite (0.16 mmoL/g), pumice stone (0.07 mmoL/g) and ceramsite (0.02 mmoL/g) (Fig. 1). As indicated in Table 1, the total pore volume of GAC and akadama clay were several orders of magnitude higher than those of other analyzed porous materials, which indicates that the oxygen-loading capabilities may be linked with their total pore volume. When the 6 oxygen-loaded materials were applied to the sediment, the improvement in the DO level were observed to follow the order of volcanic stone > zeolite > pumice stone > akadama clay > ceramsite > GAC, which revealed a distinct difference with their oxygen–loading capabilities. The difference in the oxygen-loading capability can be explained by the specific surface area and porous size distribution, while the improvement in the DO in the microcosm experiment may be more linked to the existence of mesopores (Heo and Park, 2018). An ideal oxygen carrier should possess the ability to store and transport significant amounts of oxygen while the O_2 releasing rate is under control. The investigated 6 oxygen-loaded materials showed a distinct distribution of pore size. Although the specific surface area of GAC and akadama clay (149.17 and 220.89 m^2/g, respectively, Table 1) exceeded other porous materials with the exception of ceramsite, they exhibited a predominance of larger pores (>20 nm) and smaller pores (<2 nm) (Table 1), which may either facilitate the "bubbling off" (Fig. S4) or cause the locking of oxygen. The large pore size produced a loose arrangement with oxygen bubbles, which is highly beneficial for fast oxygen transfer, and cause a quick release and bubbling off during the early stage of the releasing process, as suggested by the drastic decrease in DO concentration in the presence of oxygen-loaded GAC in the microcosm experiment 1 (Fig. 1). This "bubble off" enables the release of larger oxygen bubbles to the aqueous phase and may eventually reduce the biologically available oxygen (Lee et al., 2014). This result was consistent with a previous study, which indicated that an ion with a small radius (e.g., oxygen) that fills a large space can produce a loose arrangement with larger voids, thus cause a fast release (Işıklan, 2007). The highest value for oxygen-loading capability by GAC may also be attributed to the enhanced interaction between O_2 and the adsorbent in these narrow micropores (pore size less than 2 nm), during which the tight arrangement with smaller pores may occur hence substantially reduce the oxygen releasing rates. Consequently, volcanic stone, which comparatively possessed a higher specific surface area and pore volume, but the prevalence of mid-sized mescopores of 2–10 nm (Table 1), helped depressing the drastic release of loaded oxygen bubbles, resulting in slow release of the smaller oxygen bubbles in the water phase and benefited the improvement in the DO concentrations.

In addition to the fact that the pore size of the porous materials exhibits a close relationship with the oxygen-releasing efficiency, the affinity of the oxygen towards a specific adsorbate may also affect the oxygen-loading and the oxygen-releasing performance (Plaza et al., 2014), which explains the discrepancy between the oxygen-loading capability and DO improve efficiency. A description of the linkage between the O_2 affinity to the adsorbents and oxygen-releasing efficiency requires further detailed studies. Nevertheless, this study provides a potential method for improving the DO concentration using an ecological approach.

4.2. Oxygen-loaded porous materials are effective for short-term anaerobic modulation

This study presents a simple method for increasing the oxygen level using porous materials as oxygen carrier. The results demonstrate that the material is effective for delivering oxygen to the sediment (Fig. 4) and aqueous phase (Fig. 2). In this study, the majority of the investigated porous materials exhibited an oxygen-loading capability despite the variation in loading quantity (Fig. 1). When the oxygen-loaded porous materials were applied to the water column, the materials capped on the surface sediment by gravity settling and required minimum energy consumption. Oxygen bubbles were also observed in the sediment surface when the oxygen-loaded porous materials were used as the capping layer (Fig. 5c), which indicates that they can be diffused to the surrounding water and contributed to the increase in DO concentration. Therefore, the addition of the oxygen-loaded porous materials increased the DO concentration from 0.20 mg/L to 2.4–11.7 mg/L in the short term (Fig. 2). The retention of oxygen in the porous material facilitated the diffusion of oxygen bubbles to sediment (Fig. 3), as revealed by the considerable increase in the DO concentration across the sediment core after an 11-day incubation (Fig. 3), which suggests that the oxygen carried by the porous material can penetrate into the sediment.

The elevated DO concentration in the anaerobic sediment and water is of vital importance to the restoration of the catastrophic aquatic ecosystem. This study showed that the concentration of DO considerably increased when the oxygen-loaded material was added to the system, however, after 0.5–9 days of application, the O_2 concentrations hardly increased with time, with the exception of the oxygen-loaded volcanic stone treatment, which sustainably released oxygen during the 11 days and maintained nearly 7.80 mg/L at the end of the microcosm experiment 1 (Fig. 2). In some in situ applications, the addition of CaO_2 can increase the DO level to a high level that exceeded the demand of oxygenation and contaminants degradation, and was always associated with the exhaustion of oxygen production in several hours or days (Lee et al., 2014; Wang et al., 2016). Compared with a recent study that determined the released oxygen from granulated CaO_2 lasted for 40 weeks in a pond experiment (Nykänen et al., 2012), the oxygen improvement using porous materials was observed in a shorter time. Nevertheless, it is promising to apply oxygen-loaded porous materials in the environment, considering that the porous material possesses many distinctive properties, such as environmental harmless and reasonable environmental cost, compared with other chemicals or mechanical aeration technology when employed as an oxygen carrier.

4.3. Combined efforts of oxygen-loaded porous material and submerged macrophyte caused sustainable DO improvement

The oxygen releasing capacity of the porous materials during a long period is critical, which enables the continuous oxygenation of the water bodies by supplying O_2 for microbes and improvement in water quality (Lu et al., 2017). In our microcosm study 1, the oxygen was almost totally released from the porous material in 9 days, with the exception of volcanic stone (Fig. 2), which indicates that the

modulation of anaerobic sediment may be impaired. Although volcanic stone exhibited a lower oxygen adsorbing capability than GAC (Fig. 1), the improvement in DO is the most distinct among the analyzed materials. However, volcanic stone that carried oxygen can be completely consumed with time (probably >11 days is required). To extend the release period of oxygen, different methods have been developed. Modifications of the chemicals with other materials were made to control the O_2 releasing rate. For example, encapsulation of CaO_2 in beads were demonstrated to substantially improve the performance with slow oxygen release and constant pH maintenance (Lee et al., 2014). In the previously mentioned modifications, the added CaO_2 significantly increased the pH and influenced the subsequent bioremediation process (Zhou et al., 2017). Therefore, the development of chemical free methods for anaerobic sediment modulation is necessary.

Submerged macrophytes, which are capable of preventing sediment resuspension and increasing primary productions in aquatic systems, are a key factor in maintaining a clear state in eutrophic waters (Wang et al., 2017a). The limiting factors for the growth of submerged macrophytes include light, temperature and oxygen level (Son et al., 2018), of which light and DO are always depleted in malodorous black water. In our microcosm study 2, the oxygen released by the oxygen-loaded porous material substantially mitigated sediment anoxia and provided favorable conditions for the submerged macrophytes to growth (Fig. 5 c, d and f). Capping the surface sediment is also important for short time improvement of DO; concomitantly, the elevated DO level provides a window period to promote the growth and restoration of submerged vegetation, and thus, stabilizing the lake ecosystem can be achieved by numerous positive feedback mechanisms (Liu et al., 2018). This conclusion can be supported both by the largely increased submerged macrophytes biomass (Fig. 5f, from initial 2.20 g to final 7.81 g) and the failure of sustainable improvement of the DO level when only submerged macrophytes were incubated (Fig. 4a). Our result was also in accordance with previous study, which determined that the metabolism of living organisms and seed germination can be enhanced in the presence of air bubbles (Owusu-Ansah and Banerjee, 2009). As illustrated in Fig. 4a, compared with submerged macrophytes incubated or oxygen-loaded porous material amended treatments, the combined use of submerged macrophytes and oxygen-loaded porous material triggered oxygen levels up to 12 mg/L during the 41 days incubation, and the DO level was maintained at 7.0–8.0 mg/L towards the end of the test, which indicates the reversal of the malodorous black tide and the restoration of the aquatic ecosystems.

4.4. Manipulation of water quality by the interactive effect of oxygen-loaded material and submerged macrophyte

With the accelerated process of urbanization, the problems of malodorous black water are becoming more critical and have currently evolved into one of the main environmental issues. Oxygen depletion is a common state of these malodorous black waters and poses a potential threat to benthic inhabitants and aquatic ecosystems (Nykänen et al., 2012). Therefore, restoring the DO level in water is essential for the remediation of polluted malodorous black water (Zhou et al., 2017). In our laboratory tests, application of oxygen-loaded porous substantially improved the DO concentration and the effects were substantially manifested with the co-efforts of submerged macrophyte, during which the oxygen-loaded porous largely provided favorable condition for the growth of submerged macrophyte. The latter can sustainably raise the DO level through photosynthesis. As a result, the DO levels were maintained at above 4 mg/L throughout the 41-day experiment (Fig. 4a).

The depletion of oxygen was responsible for the disappearance of aquatic species, water eutrophication, denitrification of nitrogen (Forth et al., 2014), and production of undesirable greenhouse or malodorous gases (Liikanen and Martikainen, 2003; Miao et al., 2006). With a long-lasting improvement in DO concentrations, the production of CH_4 was reduced by 85.82% in oxygen-loaded porous material capping followed by submerged macrophyte addition treatments compared to the control (Fig. 4c), which implies the depression of the anaerobic methanogenesis. The DO levels can also affect nitrogen transformation. The nitrogen removal process particularly nitrification (which was the first and rate-limiting process for nitrogen transformation), was largely limited by insufficient DO. Capping the surface sediment with oxygen-loaded volcanic stone may contribute to the reduction of NH_4–N via adsorption and nitrification, whereas a 1.33-fold higher NH_4–N flux compared to the control was attained during the 41-day incubation. This result demonstrated the weak effects of the oxygen-loaded material in the long-term combating of the anaerobic sediment, which implies that the pristine volcanic stone may rarely induce a block effect towards NH_4^+ release. In addition, the amendment of pristine volcanic stone following the incubation of submerged macrophyte did not exhibit an improvement in the water quality because the macrophyte was quickly decomposed after 7 days of incubation (Fig. S3). With the joint efforts of the oxygen-loaded porous material and submerged macrophyte, the DO level at the SWI exhibited significant increase (Fig. 4a) which benefited the NH_4–N reduction by nitrification (Brzozowska and Gawrońska, 2009), and under the combined efforts of submerged macrophyte assimilation, a negative NH_4–N flux across SWI was observed after 41 days investigation (Fig. 4b).

The biological remineralization of organic matter consumes vast quantities of O_2 in sediment; thus, it is the most crucial cause of anoxia in waters (Nykänen et al., 2012). The exhaustion of oxygen can affect carbon transformation processes and facilitate CH_4 productions. The release of CH_4 from the sediments was primarily controlled by the O_2 availability (Liikanen and Martikainen, 2003). Thus, when oxygen is present in overlying water and served as electron acceptors, aerobic microbial processes, including CH_4 oxidation, may dominate in the surface sediments. This process likely prompted the CO_2 production. However, depressed CO_2 flux (Fig. 4d) and TIC content (Fig. 5a) were recorded in our study, probably due to the fact that 1) the CO_2 was converted in to HCO_3^-, which caused the reduction of pH (as shown in Fig. S5, the pH was maintained at nearly 8.01 in the oxygen-loaded material capping plus submerged macrophyte addition systems, which indicates a more tender effect on pH, while reaching 9.25 in the control), and 2) the inorganic carbon produced by the mineralization of organic matter was constricted in the water column, but then assimilated by the submerged macrophyte, as revealed in Fig. 5b, c, d and f. Given the above data and discussions, the combined employment of oxygen-loaded porous material and submerged macrophyte triggered multiple ecological responses in malodorous black water, including the sustainable improvement of DO concentration, promotion of the remineralization of organic matter, depression of anaerobic methanogenesis meanwhile enhancement of CO_2 production, regulation of nitrogen flux across SWI, and creation of favorable conditions for the growth of submerged macrophyte, which may in turn contributes to increase DO levels. These multiple processes are illustrated in Fig. 6.

5. Conclusions

Ecological restoration of malodorous black water is crucial in China, particularly after the implementation of pollution abatement and control programs. This study proposes a simple and eco-
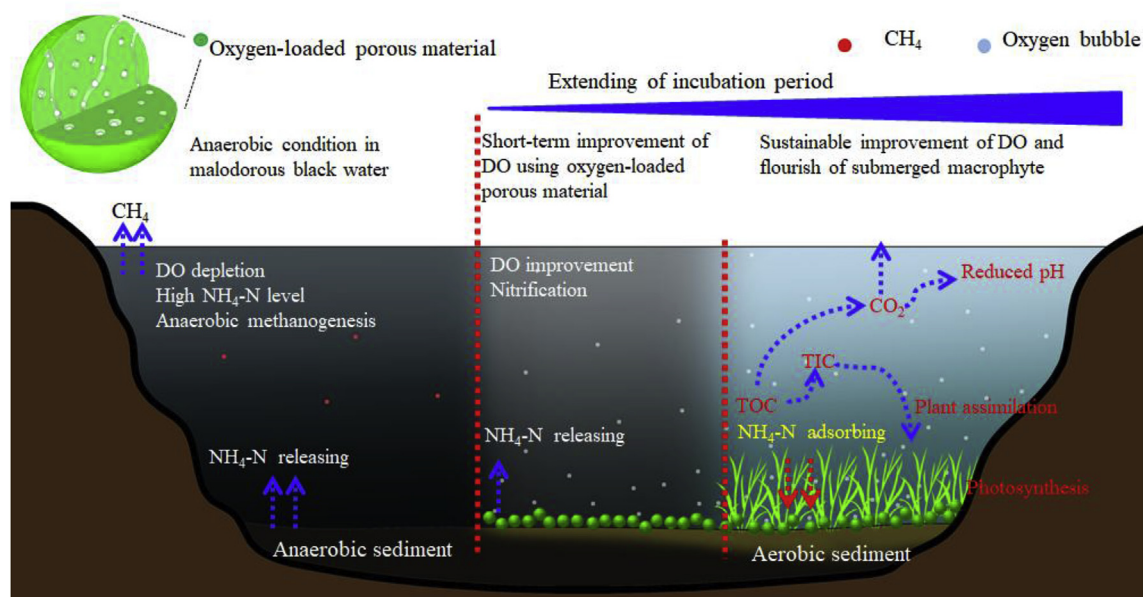
Fig. 6. Conceptual schematic of the sustainable improvement in DO levels in anaerobic sediment of malodorous black water, as well as water quality regulation, through joint efforts from oxygen-loaded volcanic stone and submerged macrophyte.

friendly approach to preparing oxygen-loaded material by the removal of air from the porous materials followed by filling them with pure O_2. Among the studied 6 investigated porous materials, the GAC possessed the highest oxygen-loading capability, while volcanic stone were most efficient in improving DO concentration at the SWI. This discrepancy can be explained by the pore size distribution, of which the GAC showed massive small (<2 nm) and large (>20 nm) pores but mid-sized pores (2–10 nm) were prevalent in volcanic stone. Capping the anaerobic sediment with oxygen-loaded volcanic stone increased the DO concentration during our 11-day experiment, but can be extended to >41 days when submerged macrophyte was added. The combined employment of oxygen-loaded volcanic stone and submerged macrophyte triggered continuous improvement of DO in malodorous black water, accompanied by the reduction of CH_4 and the depression of NH_4–N release from sediment, as well as the 2.55-fold increase in the submerged macrophyte biomass. Thus, a sustainable modulation of the investigated malodorous black water was achieved. The proposed approach can potentially help to outcompete the combat towards the restoration of malodorous black water nationwide.

Authorship contribution statement

Ming Liu: Investigation, Data curation. Yan Ran: Investigation. Xinxin Peng: Writing review & editing. Zhiqiang Zhu: Writing review & editing. Jialiang Liang: Writing review & editing. Hong Li: Writing original draft, Writing review & editing, Funding acquisition. Qiang He: Formal analysis, Writing review & editing.

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.watres.2019.05.045.

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