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生物炭对潜流人工湿地污染物去除及N_2O排放影响

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摘要: 生物炭作为一种生物质废弃物的热解产物, 逐渐被应用于受污染水体治理. 生物炭具有提高孔隙、吸附氮磷、控制温室气体排放等作用. 通过在温室内构建生物炭投加比为 40%、30%、20%、10% 和 0% 的微型潜流湿地系统(分别命名为 BW-40、BW-30、BW-20、BW-10 和 CW-K), 探究生物炭投加对湿地污染物去除及N_2O排放的影响. 结果表明, 投加生物炭可以提高出水氧化还原电位(oxidation-reduction potential, ORP), 降低电导率(conductivity, Cond), 但影响均不显著($P>0.05$). 5 组湿地系统中化学需氧量(COD)去除率均达到 90%, 但随着生物炭投加比的增加, 氨氮(NH_4^+-N)和总氮(TN)的去除效果显著提高($P<0.05$). 湿地NH_4^+-N平均去除率为(34.76 ± 14.16)% ~ (57.96 ± 10.63)%, TN 平均去除率为(70.92 ± 5.68)% ~ (80.21 ± 10.63)%. 各湿地系统N_2O的平均释放通量在 13.53 ~ 45.30 $mg\cdot(m^2\cdot d)^{-1}$之间, 生物炭投加可以通过减少亚硝态氮(NO_2^--N)累积浓度和积累时间, 实现N_2O减排, 并显著减少湿地中N_2O排放占 TN 去除的百分比($P<0.05$). 40% 的生物炭投加比可以实现 70.13% 的N_2O减排效果.

关键词: 潜流人工湿地; 生物炭; 有机污染物去除; 脱氮; N_2O排放

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Influences of Biochar on Pollutant Removal Efficiencies and Nitrous Oxide Emissions in a Subsurface Flow Constructed Wetland

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Abstract: Biochar, pyrolyzed from agricultural biomass wastes, has been widely used as an improver in wastewater treatment to regulate the oxygen distributions and microbial communities because of its extended surface area and porous structure. In addition, biochar has been shown to play a role in enhancing the porosity, adsorbing ammonium (NH_4^+-N), and reducing nitrous oxide (N_2O) emissions. In this paper, five groups of constructed microcosm wetlands (CW) were built in a greenhouse with different biochar doses of 40%, 30%, 20%, 10%, and 0% (named as BW-40, BW-30, BW-20, BW-10, and CW-K, respectively) to investigate the influences of biochar on pollutant removal efficiencies and N_2O emissions. The results showed that the concentration of effluent dissolved oxygen (DO) was less than 0.5 $mg\cdot L^{-1}$, and the pH was stable at around 7.2 in every CW. Additionally, the effluent oxidation-reduction potential (ORP) was found to have moderately increased with the increases in the quantity of biochar, and the conductivity (Cond) test results showed the opposite trend. However, the effects of biochar on DO, pH, ORP, and Cond were not significant ($P>0.05$). The chemical oxygen demand (COD) removal rates were up to 90% in all CWs. On the other hand, significantly higher removal efficiencies for NH_4^+-N and total nitrogen (TN) were found in CWs filled with biochar ($P<0.05$). The average NH_4^+-N removal rates were (57.96 ± 10.63)%, (51.12 ± 11.74)%, (48.55 ± 8.75)%, (43.95 ± 9.74)%, and (34.76 ± 14.16)% in BW-40, BW-30, BW-20, BW-10, and CW-K, respectively, while the total nitrogen (TN) average removal rates were (80.21 ± 10.63)%, (78.48 ± 5.73)%, (76.80 ± 4.20)%, (75.88 ± 5.85)%, and (70.92 ± 5.68)%, respectively. Nitrate (NO_3^--N) was not detected in the CWs for there were sufficient carbon sources and suitable denitrification environments. Moreover, the average fluxes of N_2O ranged from 13.53 $mg\cdot(m^2\cdot d)^{-1}$ to 45.30 $mg\cdot(m^2\cdot d)^{-1}$ in the experimental systems. Compared with the control, the reduction rates of N_2O in the BW-40, BW30, BW20, and BW10 were 70.13%, 68.26%, 50.83%, and 37.90%, respectively, and the ratios of N_2O emissions to the removed nitrogen in CWs with biochar were significantly lower than those in the CW without biochar. Positive correlations were observed between the N_2O fluxes and nitrite (NO_2^--N) concentrations, and the lower N_2O emissions could be attributed to the higher oxygen transfer and lower NO_2^--N accumulation rates in response to the biochar addition. These results demonstrate that biochar could be used as an amendment to strengthen the nitrogen removal and reduce the N_2O emissions in CWs.

Key words: subsurface flow constructed wetland; biochar; organic matter removal; nitrogen removal; N_2O emission

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人工湿地由于环境友好且对氮磷具备较强的净化能力，在水处理领域得到了广泛的研究和应用[1]．传统生物脱氮高度依赖于硝化，而人工湿地中因DO供应不足导致硝化过程不能有效进行，致使脱氮效率不高[2,3]．尽管研究人员在优化该过程做出持续努力，但在氮负荷为0.6～2.0 $g\cdot(m^2\cdot d)^{-1}$时，TN去除率仅为50%左右，限制了潜流湿地的应用[4,5]．有研究发现改变湿地填料可获取更高的污染物去除效果，Liu等[3]将红砖、粉煤灰砖等介质应用于人工湿地，有效改善湿地系统处理效果．Kizito等[6]的研究表明以生物炭作为填料的人工湿地对COD、NH_4^+-N、TN和总磷的去除率明显高于普通砾石填料湿地．另一方面，生物脱氮过程中不完全硝化或反硝化过程会造成N_2O累积排放．由于缺少硝化-反硝化的有利环境[7]，湿地成为一种重要的N_2O气体排放来源[8]．生物炭作为一种生物质废弃物的热解产物，具有比表面积大、芳香化程度高、稳定性强、孔隙大、质量轻等特点，具备调节好氧-缺氧环境、强化硝化-反硝化的重要作用[9,10]．Cayuela等[11]的研究表明，在14种不同的农业土壤中，生物炭投加使N_2O排放量下降了10%～90%．生物炭用于强化湿地系统脱氮和调控N_2O减排的研究正在兴起[12,13]，为此，本研究采用湿地微型系统，探讨生物炭投加对潜流人工湿地污染物去除及N_2O排放的影响，以期为生物炭在人工湿地污水处理中应用提供必要的技术支撑和理论依据．

1　材料与方法

1.1　湿地系统构建

湿地反应器采用圆筒形聚乙烯容器，每个容器表面积为0.1 m^2，深为35 cm．按照生物炭40%、30%、20%、10%和0%的添加比例分别构建5组微型系统，每组设置2个平行，共计10个湿地反应器．反应器构建按照文献[14]进行．湿地植物选用菖蒲(*Acorus calamus* L.)，植物经驯化扩培后植入湿地，栽种密度为30株$\cdot m^{-2}$，试验装置如图1所示．生物炭以湿地植物芦竹秸秆为原料，参照Huang等[9]的制备方法，于500℃条件自制获得．生物炭的比表面积为345.92 $m^2\cdot g^{-1}$，孔径为1.95 nm，孔容为0.2467 $cm^3\cdot g^{-1}$．生物炭主要元素的质量分数为碳87.19%、氢0.40%、氧10.4%、氮1.47%和硫0.54%．

1.2　系统运行

湿地进水采用自来水配置，每升进水包括390 mg $C_6H_{12}O_6$、220 mg KNO_3、75 mg NH_4Cl、200 mg

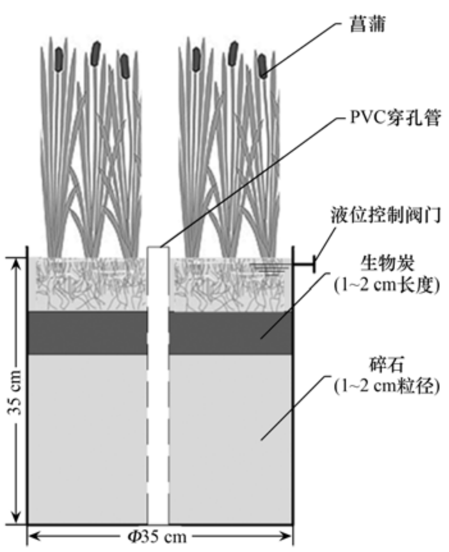

图1　试验装置示意

Fig. 1　Scheme of the experimental microcosms

$NaHCO_3$、11 mg KH_2PO_4、18 mg $K_2HPO_4\cdot 3H_2O$、10 mg $MgSO_4\cdot 7H_2O$、10 mg $FeSO_4\cdot 7H_2O$、7.6 mg $CaCl_2$和1 mL微量元素液．每升微量元素液包括0.15 g H_3BO_3、0.03 g $CuSO_4\cdot 5H_2O$、0.18 g KI、0.12 g $MnCl_2\cdot 4H_2O$、0.06 g $Na_2MoO_4\cdot 2H_2O$、0.12 g $ZnSO_4\cdot 7H_2O$、0.15 g $CoCl_2\cdot 6H_2O$和10 g EDTA-Na_2．配好的进水包括(414.1±10.5)$mg\cdot L^{-1}$ COD、(19.7±1.7)$mg\cdot L^{-1}$ NH_4^+-N、(29.8±1.2)$mg\cdot L^{-1}$硝态氮(NO_3^--N)和总磷(5.1±0.4)$mg\cdot L^{-1}$，进水pH控制为(7.5±0.3)，DO为(7.0±0.5)$mg\cdot L^{-1}$．湿地孔隙率为(47.2±0.6)%～(50.4±1.2)%，水力停留时间为2 d，处理负荷设定为0.05 $m^3\cdot(m^2\cdot d)^{-1}$，有效进水量为10 L，进水水位由顶部水位控制阀门调整．

1.3　样品采集与测定

1.3.1　水质测定分析

系统稳定运行期间每2d进行一次水质测定，其中COD、NH_4^+-N、NO_3^--N、NO_2^--N和TN均按国标法进行测定[15]，DO、pH、ORP和Cond(conductivity，电导率)分别采用便携式溶氧仪(Pro ODO，YSI，美国)、便携式pH计(PB-10，Sartorius，德国)、便携式ORP测定仪(PH100，YSI，美国)和便携式电导率仪(DDS-307，上海右一，中国)测定．

1.3.2　N_2O气样采集与测定

N_2O通量采用静态箱-气相色谱法测定[16]．静态箱由有机玻璃制成，表面采用铝箔包裹防止光辐射．箱体高1.2 m，直径0.3 m，内部交错安装3个风扇，用于混匀箱内气体，内壁挂装有大气压计，用于测定箱内气压和温度．采集气体时，将静态箱罩在湿地表面，底部水封．气样在水质稳定周期内

采集，每周采集一次，共计 5 次．每个监测周期以进水时刻为起点设定 7 个采样时间节点(0、3、6、9、12、24、36 和 48 h)，每个时间节点持续采样 1 h，即罩静态箱后的 0、10、20、40 和 60 min 采集气体，并同时记录箱内温度，气压．采用气相色谱仪(Agilent 7820A，USA)测定 N_2O 浓度．仪器配有 μECD 检测器和 Porapak Q 填充柱．ECD 检测器、进样口和色谱柱的温度分别设定为 300、100 和 70℃，以 99.999% 高纯 N_2 为载气，载气流速 30 $mL\cdot min^{-1}$．

N_2O 通量采用如下公式[16]计算：

$$J = \frac{dc}{dt} \cdot \frac{M}{V_0} \cdot \frac{P}{P_0} \cdot \frac{T_0}{T} \cdot H$$

式中，J 为 N_2O 释放通量，$mg\cdot(m^2\cdot h)^{-1}$；dc/dt 为静态箱内 N_2O 浓度随时间的变化率，$mm^3\cdot(m^3\cdot h)^{-1}$；$M$ 为 N_2O 的摩尔质量，44.02 $g\cdot mol^{-1}$；P 为静态箱中的气压，Pa；T 为静态箱中的热力学温度，K；V_0、P_0 和 T_0 分别为标况下的气体摩尔体积($L\cdot mol^{-1}$)、气压(Pa)和温度(K)；H 为水面以上静态箱高度，m．

1.4 数据分析

全部样品水质和气体分析测试设置平行 3 次，数据表达采用平均值 ± 标准差．试验数据通过 Origin 8.5 整理作图，并由 PASW Statistics 18.0 进行数据分析．对象之间相互关系采用相关性分析，并经 Pearson 检验(水平包括显著 $P < 0.05$ 和极显著 $P < 0.01$)．对象之间的差异性分析采用 One-way ANOVA (水平包括显著 $P < 0.05$ 和极显著 $P < 0.01$)．

2 结果与讨论

2.1 不同生物炭湿地出水中 DO、pH、ORP 和 Cond 变化

如图 2 所示，5 组湿地系统运行 30 d 后，出水 DO 逐渐趋于稳定，随着湿地运行时间增加，各系统出水 DO 逐渐下降至 0.2 $mg\cdot L^{-1}$ 左右，这一结果与 Lyu 等[17]的研究结果一致．随着反应器运行时间增长，湿地系统中微生物大量繁殖，微生物降解污染物使耗氧量增加，导致出水 DO 降低．各反应器中出水 pH 值稳定在 7.0 ~ 7.5 范围，ORP 值在(−400 ~ −350 mV)之间，随着湿地运行时间增长，系统还原性逐渐增强．湿地稳定运行后，BW-40 中 ORP 值高于对照湿地，而 Cond 值较对照湿地低，生物炭添加能在一定程度上增加 ORP，强化硝化过程和部分缺氧代谢，降低出水 Cond[18]．多孔生物炭填料有利于氧的传递、减缓湿地还原性强度，改变了湿地微环境，但生物炭投加对湿地出水 DO、pH、ORP 和 Cond 影响均不显著($P > 0.05$)．

2.2 湿地有机污染物去除及脱氮效果

不同生物炭投加比例下湿地系统对 COD 的去

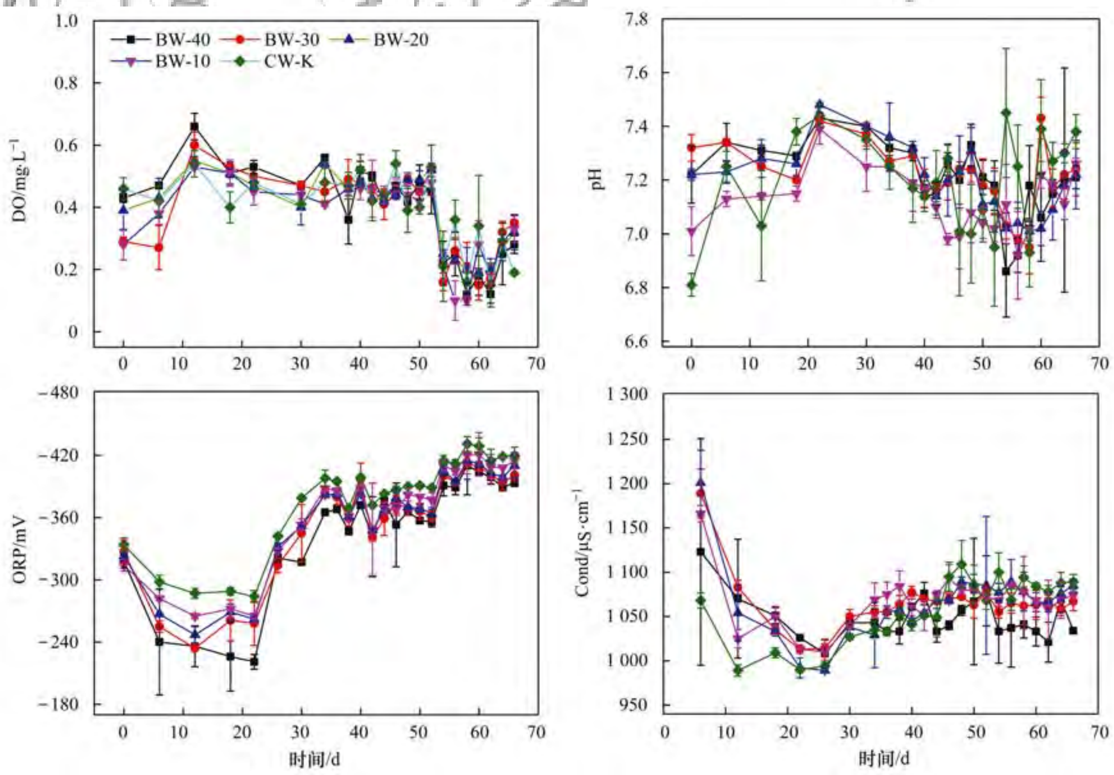

图 2 湿地系统出水 DO、pH、ORP 和 Cond

Fig. 2 The pH, DO, ORP, and Cond in the effluent of every CW

除率均达到90%（图3）. 在湿地系统中，有机物主要通过好氧、厌氧生物代谢和有机物沉淀等作用去除[19, 20]. 系统出水COD浓度随着生物炭投加量增加而逐渐降低，生物炭投加对COD去除起到了一定促进作用，但影响不显著（$P>0.05$）. 但生物炭投加显著提升湿地对NH_4^+-N去除效果（$P<0.05$），出水NH_4^+-N平均浓度在（8.28 ± 2.27）~（12.86 ± 3.19）$mg \cdot L^{-1}$之间，CW-K ~ BW-40 对NH_4^+-N的平均去除率依次为（34.76 ± 14.16）%、（43.95 ± 9.74）%、（48.55 ± 8.75）%、（51.12 ± 11.74）%和（57.96 ± 10.63）%. 湿地系统中生物炭通过两个主要途径促进NH_4^+-N去除，一是多孔松散的结构为氧气扩散进入湿地床体提供条件，有利于硝化细菌生长并促进硝化作用，使生物炭湿地具备良好的脱氮效果；二是生物炭强化NH_4^+-N吸附作用，导致出水

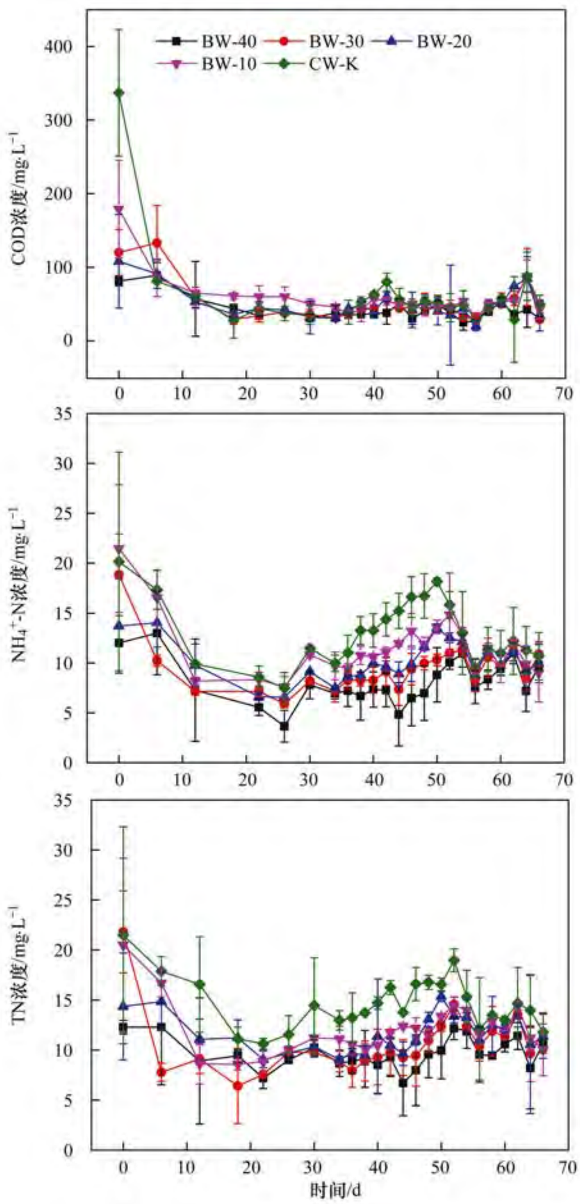

图3　湿地系统中COD、NH_4^+-N和TN的去除效果

Fig. 3　Removal efficiencies of COD, NH_4^+-N, and TN in CWs

NH_4^+-N浓度降低[21~23]. 随湿地运行时间增长，生物炭吸附能力达到饱和，且微生物大量富集减小了生物炭填料对氧气的扩散作用，导致生物炭湿地长期运行后NH_4^+-N去除效果有所下降[22]，但生物炭湿地NH_4^+-N去除效果仍优于对照湿地.

各湿地系统（CW-K ~ BW-40）对TN的平均去除率依次为（70.92 ± 5.68）%、（75.88 ± 5.85）%、（76.80 ± 4.20）%、（78.48 ± 5.73）%和（80.21 ± 10.63）%，生物炭投加明显提高湿地TN的去除效率（$P<0.05$）. 各反应器出水NO_3^--N均未被检出，湿地对TN的去除规律与NH_4^+-N去除相似. 湿地微型系统长期处于缺氧和充足的碳源条件下，反硝化性能强，NO_3^--N几乎被全部去除. 因此，生物炭湿地中TN去除特征与传统湿地相似，TN的完全性去除依赖于完全的硝化作用[24, 25].

2.3　污染物与N_2O的典型周期变化

典型周期内污染物变化与N_2O通量变化如图4所示，5组人工湿地DO和COD浓度随时间变化规律类似，在进水后的1 h内DO浓度迅速降至1.0 $mg \cdot L^{-1}$左右，随后缓慢降低至0.5 $mg \cdot L^{-1}$以下；而COD基本在进水后6h内降到50 $mg \cdot L^{-1}$左右，去除率达85%以上，生物炭添加对COD降解速率影响不显著（$P>0.05$）. 湿地中NH_4^+-N大部分在12 h内被去除，生物炭湿地中NO_3^--N在3 h内全部去除，而对照湿地中NO_3^--N在进水后3 h后仍未去除完全，原因可能是生物炭显著提高了湿地系统微生物丰度，促进反硝化过程[26]. 5种湿地微型系统中，NO_2^--N在BW-40、BW-30和BW-20中第1h时出现积累，BW-10中积累时间持续至3 h，CW-K中持续时间更长. 由此可见，生物炭投加可加速湿地中NO_3^--N的去除，并改善NO_2^--N积累情况.

生物炭湿地中（BW-40、BW-30和BW-20）N_2O排放主要集中在开始的6 h内，而BW-10和CW-K除在开始的6 h内排放N_2O外，后续的36 h仍保持较高的排放通量，并且在第12 h出现第二个N_2O排放通量峰值. 进水初期湿地系统中NH_4^+-N和DO浓度较高，NH_4^+-N被氨氧化菌（AOB）氧化成NO_2^--N和NO_3^--N，而系统中DO在前1 h内迅速下降，充足的有机物使异养型好氧微生物与自养型AOB竞争DO，限制了硝化过程，导致NO_2^--N累积[21]. 在生物炭湿地中，高孔隙率的生物炭利于空气扩散进入湿地床体，改善了系统DO和还原性环境，使得NO_2^--N积累时间明显低于对照湿地. 根据相关性分析，湿地中N_2O释放通量变化与NO_2^--N的浓度变化呈显著正相关（BW-40 ~ CW-K的相关系数分别为

0.946、0.921、0.876、0.951 和 0.822，$P < 0.05$）. 对照湿地中N_2O持续排放并出现第二个峰值的原因可能是系统低浓度的NO_2^--N持续积累，微生物为避免NO_2^--N在细胞内累积，诱发产生异构亚硝酸还原酶，使NO_2^--N被还原产生大量的N_2O[27].

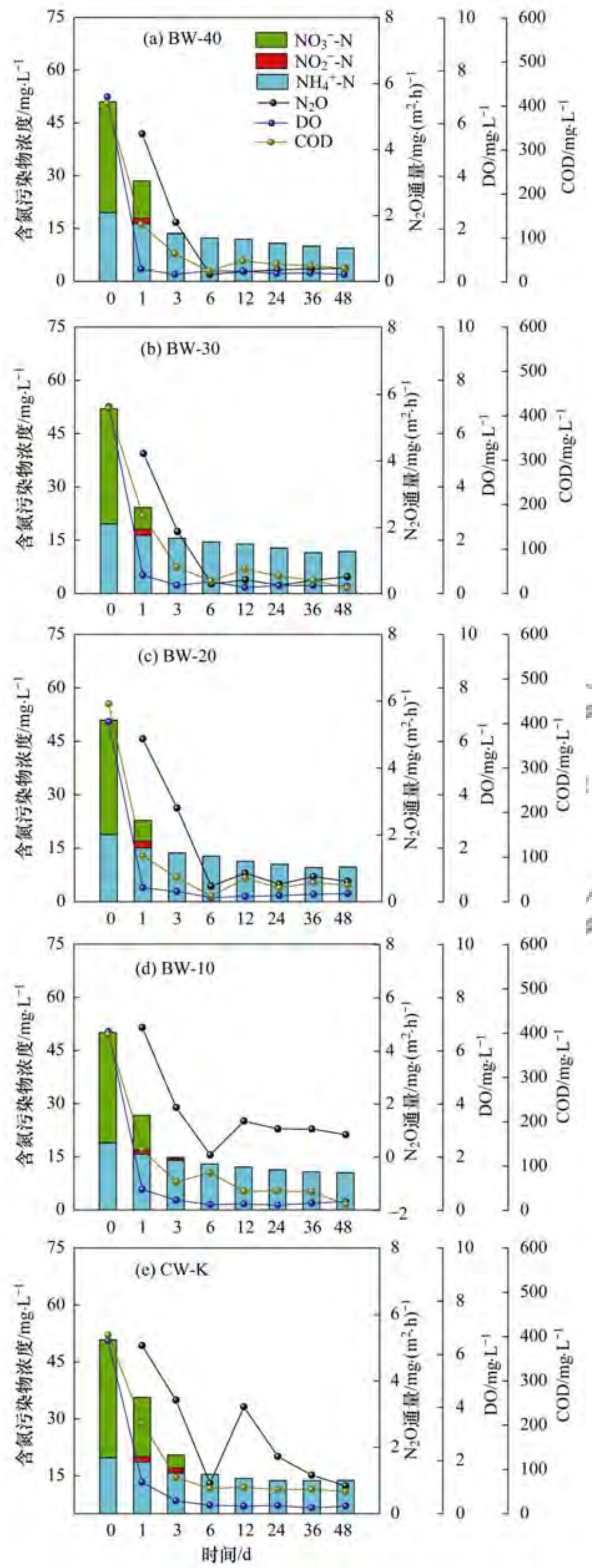

图 4 典型周期内NH_4^+-N、NO_3^--N、NO_2^--N、DO、COD 和N_2O通量动态变化

Fig. 4 Dynamic changes of NH_4^+-N, NO_3^--N, NO_2^--N, DO, COD, and N_2O emissions in a typical operating period

2.4 不同生物炭湿地中N_2O释放特征

湿地中N_2O累积量如图 5 所示，5 组湿地周期内N_2O累积释放量在 27.06 ~ 90.59 $mg \cdot m^{-2}$之间，以周期(48 h)内释放的N_2O计算湿地 BW-40 ~ CW-K 中N_2O日平均排放通量分别为 13.53、14.38、22.27、28.13 和 45.30 $mg \cdot (m^2 \cdot d)^{-1}$. Wu 等[16]研究不同碳氮比条件下湿地$N_2O$排放通量在 0.03 ~ 134.2 $mg \cdot (m^2 \cdot d)^{-1}$与本研究结果一致. 生物炭投加显著减少湿地系统$N_2O$累积释放量，BW-40 ~ BW-10 与对照湿地相比分别实现N_2O减排 70.13%、68.26%、50.83% 和 37.90%.

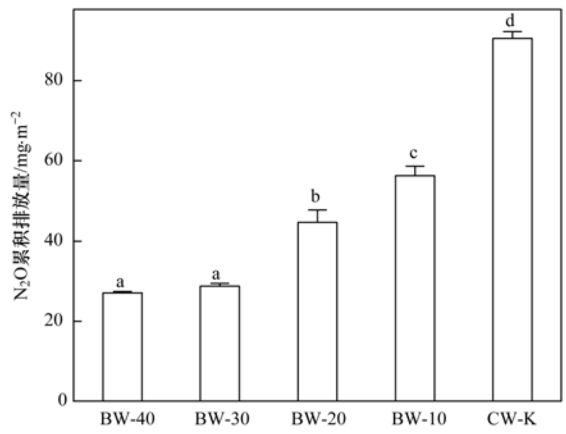

图 5 湿地中N_2O累积释放量

Fig. 5 Accumulation emissions of N_2O in different CWs

生物炭投加同时也可以显著降低湿地中以N_2O排放脱氮占 TN 去除的比例(图 6)，在对照湿地中，N_2O排放占到 TN 去除的 1.5% 以上，该占比与 Li 等[25]的研究结果(1.44%)和 Zhang 等[28]的研究结果(1.0% ~2.1%)一致. 随着生物炭投加量增加，

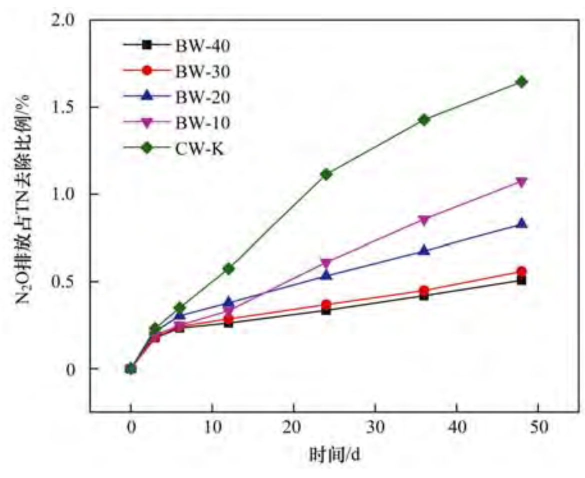

图 6 不同湿地中N_2O排放占 TN 去除比例(以氮计)

Fig. 6 Ratios of N_2O emissions to TN removal in different CWs (calculated with nitrogen)

N_2O排放量占 TN 去除的比例逐渐下降，其中 10% 的生物炭能使占比降至 1% 以下，而 40% 和 30% 的生物炭能够使占比下降至 0.5% 以下.

生物脱氮过程中，N_2O主要作为硝化过程的副产物及反硝化过程的中间产物产生. 生物炭可作为潜在碳源，促进反硝化过程并减缓系统 pH 值变化、促进电子向反硝化微生物转移、促进N_2O还原为 N_2，减少N_2O排放[11, 29]. 湿地环境对反硝化作用极为有利，充足的碳源和较低 DO 水平使得湿地系统不具备反硝化作用产生N_2O的有利条件，反硝化过程对湿地N_2O排放的贡献较小[30]，硝化过程便成为控制湿地系统N_2O排放的关键. 从污染物与N_2O的典型周期变化分析可知，硝化过程中NO_2^--N积累是导致N_2O产生的重要因素. 生物炭对缓解湿地系统中NO_2^--N积累起重要作用，实现N_2O减排.

3　结论

(1)在潜流人工湿地中，生物炭投加对有机物的去除影响不显著，但可以显著提升氨氮和总氮的去除能力，高生物炭投加比例能实现较高的脱氮效果.

(2)生物炭对人工湿地中N_2O排放的控制作用明显，10% ~40% 的生物炭投加可实现N_2O减排 37.90% ~70.13%，并显著减少N_2O排放占 TN 去除的百分比.

(3)湿地系统中NO_2^--N累积与N_2O排放密切相关，生物炭投加可以通过减少NO_2^--N累积实现N_2O减排.

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