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# Adsorption of phosphate and mitigation of biofouling using lanthanum-doped quorum quenching beads in MBR

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**Abstract.** The removal of phosphorus, especially phosphate-form phosphorus, is necessary in wastewater treatment. Biofouling induced by the quorum sensing mechanism is also a major problem in membrane bioreactor (MBR), which reduces membrane flux. This study introduces lanthanum-doped quorum quenching (QQ) beads into MBR, confirming their inhibitory effect on biofouling due to *Rhodococcus sp. BH4* and their capacity for phosphorus removal through lanthanum adsorption. A batch test was conducted to access the phosphate adsorption of lanthanum-QQ (La-QQ) beads and lab-scale MBR to verify the effect of inhibition. The study aimed to identify distinctions among the MBR, QQ MBR, and La-QQ MBR. In the batch test, the phosphate removal rate increased as the volume of beads increased, while the unit volume removal rate of phosphate decreased. In the lab-scale MBR, the phosphate removal rates were below 20% in the control MBR and QQ MBR, whereas the La-QQ MBR achieved a phosphate removal rate of 74%. There was not much difference between the ammonia and total organic carbon (TOC) removal rates. Regarding the change in transmembrane pressure(TMP), 3.7 days were taken for the control MBR to reach critical pressure. In contrast, the QQ-MBR took 9.8 days, and the La-QQ MBR took 6.1 days, which confirms the delay in biofouling. It is expected that La-QQ can be used within MBR to design a more stable MBR process that regulates biofouling and enhances phosphate removal.

**Keywords:** adsorption; biofouling; lanthanum beads; MBR; phosphate removal; quorum quenching

## 1. Introduction

The excessive discharge of nutrients into aquatic environments can lead to severe eutrophication, posing a threat to natural ecosystems by triggering harmful algal blooms (Wang *et al.* 2021a, Zhang *et al.* 2024). Specifically, phosphorus (P), an essential element for living organisms, is a major cause of eutrophication in aquatic ecosystems (Zong *et al.* 2022). Therefore, the removal of phosphorus is inevitable, and various phosphorus removal methods exist, including coagulation, adsorption, and biological treatment (Chu *et al.* 2018). Among these methods, the coagulation process is widely used because of its high removal efficiency and ease of operation (Wang *et al.* 2022, 2021b). However, it has drawbacks such as land consumption and maintenance costs because a separate reactor must be installed after biological treatment process to establish a dedicated process for phosphorus removal (Bashar *et al.* 2018). Total phosphorus (T-P) mainly exists in the form of phosphate and particulate phosphorus (Zeng *et al.* 2004). Although, particulate phosphorus can be removed through solid precipitation in secondary clarifier or membrane separation processes. However, the removal of phosphate-form phosphorus requires a separate process (Hasbullah *et al.* 2020, Pang *et al.* 2016). In particular, coagulation processes involving metals such as iron or aluminum have received attention. Recent research, however, has highlighted the importance of lanthanum-based adsorbents. Lanthanum is chemically similar to iron or aluminum and remains stable over a wide pH range, thereby enhancing the importance of lanthanum-based adsorbents (Lu *et al.* 2020)

Membrane bioreactor (MBR) processes are used as alternatives to conventional physical-chemical and biological treatment processes in the field of wastewater treatment, offering high treatment efficiency and stable effluent quality (Sameer *et al.* 2021, Kim *et al.* 2013, Ahmed *et al.* 2020). However, MBRs have a critical weakness in the form of ongoing biofouling, where the membrane pores become clogged owing to biofilm formation on the membrane surface (Jiang *et al.* 2021, Bin *et al.* 2008). To deal with this problem, repeated cleaning with high-concentration chemical agents is required, leading to a deterioration in membrane performance.

Occurrence of membrane fouling results from microbial quorum sensing (QS) mechanisms (Perveen. 2018, Lee *et al.* 2018). Involving quorum quenching (QQ) mechanism is one method to inhibit this circumstance (Weerasekara *et al.* 2016). Disrupting microbial signal generation and reception or deactivating N-acyl homoserine lactone (AHL) signaling molecules suppresses extracellular polymeric substance

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Table 1 Crosslinking conditions for making different types of bead

| Sol. Types | PVA[1)] (%) | SA[2)] (%) | CL[3)] solutions | | CL reaction time(hours) | |
|---|---|---|---|---|---|---|
| | | | 1st | 2nd | 1st | 2nd |
| Bead #1 | 8 | 1 | 4% $CaCl_2$ + 7% Boric acid | 0.5 M $Na_2SO_4$ | 0.5 | 2 |
| Bead #2 | 8 | 1 | 4% $CaCl_2$ + 7% Boric acid | 0.5 M $Na_2SO_4$ | 2 | 4 |
| Bead #3 | 8 | 1 | 6% $LaCl_3$ + 7% Boric acid | 0.5 M $Na_2SO_4$ | 0.5 | 2 |
| Bead #4 | 8 | 1 | 6% $LaCl_3$ + 7% Boric acid | 0.5 M $Na_2SO_4$ | 2 | 4 |

1) Polyvinyl Alcohol, 2) Sodium Alginate, 3) Cross-Linking

(EPS) production which delays membrane biofouling (Nahm *et al*. 2017, Huang *et al*. 2016, Lee *et al*. 2016, Taskan *et al*. 2021, Song *et al*. 2023). QQ mechanism of *Rhodococcus sp*. *BH4* microorganisms is utilized to inhibit QS through inactivation of AHL molecules, and since *Rhodococcus sp*. *BH4* is a microorganism that also produces EPS, which can be utilized by immobilizing it on beads (Nam *et al*. 2015). Various previous studies have been conducted using polyvinyl alcohol-sodium alginate (PVA-alginate) beads inoculated with QQ microorganisms and circulated within the reactor to inhibit biofouling of the membrane (Lee *et al*. 2016b).

Therefore, this study aimed to address issues regarding T-P concentration in the treated water and membrane fouling in the MBR process using QQ beads. The goal was to achieve stable effluent quality and delay membrane fouling in the MBR process by employing QQ beads. Lanthanum-doped QQ (La-QQ) beads were fabricated to delay membrane biofouling through QQ mechanisms and overcome the drawbacks of the MBR process by adsorbing phosphates with lanthanum ions. This study was conducted to mitigate the challenges associated with T-P concentration and membrane fouling in MBR systems.

## 2. Materials and methods

### 2.1 Preparation of La-QQ beads

PVA-alginate beads were fabricated as detailed in Table 1. Polyvinyl alcohol (PVA) 8% w/v and sodium alginate (SA) 1% w/v were dissolved in distilled water, the mixture was then sufficiently dissolved by heating to 105℃ on a hot plate while stirring for 4 hours. The uniformly dissolved PVA-alginate solution was cooled down to 40 ℃. Using a pump, nozzle, and syringe with pressure control, the solution was slowly injected into the first cross-linking solution. Subsequently, we performed a washing process using distilled water, followed by the second cross-linking step. The beads were then placed in deionized water and stored at 4 ℃ (Islam *et al*. 2022, Islam *et al*. 2020).

SEM-EDS (EM-30AX, COXEM, Korea) was used to evaluate the extent of lanthanum doping on the surface of the manufactured beads and to analyze the surface morphology and lanthanum distributions (Ayarza *et al*. 2016). QQ bacteria *Rhodococcus sp*. *BH4* (Oh *et al*. 2013) was employed to induce QQ mechanisms within the membrane reactor. *Rhodococcus sp*. *BH4* was inoculated into Luria-Bertani (LB) broth for proliferation. After growth, the culture was transferred to a 50 mL conical tube and centrifuged at 4000 rpm for 30 minutes to obtain a pellet, which was then resuspended in 10 mL of deionized water. The resulting deionized water containing *Rhodococcus sp*. *BH4* was thoroughly mixed with the PVA-alginate solution, achieving a concentration of 5 mg *BH4* per 1 mL. Using this solution, beads were prepared according to a previously described method (Kim *et al*. 2023).

### 2.2 Adsorption of phosphate by La-QQ beads

Batch experiments were conducted to evaluate the extent of lanthanum-facilitated phosphate adsorption on the surface of the beads. These experiments were performed using a jar tester, and the cases were set up as shown in Table 2. Each jar was filled with a 400 mL solution containing 10.0 mg/L as P, and the appropriate beads for each case were added. The mixtures were stirred at 150 rpm for 24 hours to conduct the adsorption experiments. After completion of the batch experiments, the T-P concentration in each jar was analysed by using T-P analysis kit (T-P Kit Low Range/27426-45, Hach) and a spectrophotometer (DR 6000, Hach).

### 2.3 Operation of the lab-scale MBR experiment

The lab-scale MBR system shown in Fig. 1, was configured for automatic control and operation via a system embedded in the programmable logic controller (PLC) panel. The MBR process was operated in cycles of 10 minutes of filtration followed by 1 minute of pause. Operational conditions included mixed liquor suspended solids (MLSS) maintained at 8,000 mg/L and dissolved oxygen (DO) levels below 0.1 mg/L during the anoxic phase and between 2.0~2.5 mg/L during the aerobic phase. The flux was set at 25 Lm-2 h-1, with other operating conditions detailed in Table 3. The microfiltration membrane (MEMBRIO, LOTTE Chemical Corp.) used in the lab-scale MBR was made of polyvinylidene fluoride (PVDF) material. The hollow fiber of membrane has 0.03μm of pore size, and outer and inner diameter were 2.1 mm and 0.8 mm, respectively.

Operational data for the MBR process were automatically logged at 5-minute intervals. The operation of MBR was terminated when the logged transmembrane pressure (TMP) data reached to 0.5 bar. Synthetic wastewater was used for the experiment, which was formulated based on the average

Table 2 Case of the adsorption phosphate batch test

| Case No. | Numbers of beads in a jar (#) | Bead volume ($cm^3$) |
|---|---|---|
| Case 1 | 0 | 0 |
| Case 2 | 40 | 0.804 |
| Case 3 | 80 | 1.608 |
| Case 4 | 120 | 2.413 |
| Case 5 | 160 | 3.217 |
| Case 6 | 200 | 4.021 |
| Case 7 | 240 | 4.825 |

Table 3 Operating conditions of the lab-scale MBR

| Parameters | Operation values |
|---|---|
| MLSS (mg/L) | 8,000 |
| DO in the aerobic reactor (mg/L) | 2.0~2.5 |
| DO in the anoxic reactor (mg/L) | ≤ 0.1 |
| Aeration rate (L/min) | 1.0 ~ 1.5 |
| Hydraulic retention time (hrs) | 3.5 ~ 5.0 |
| Flux ($L/m^2$ h) | 25 |
| Filtration cycle | Filtration : 10 min -Pause : 1 min |

Table 4 Characteristics of influent water quality of lab-scale MBR

| Items | Domestic wastewater | Synthetic wastewater |
|---|---|---|
| $BOD_5$ (mg/L) | 200.4 | 171.6 |
| $COD_{Mn}$ (mg/L) | 170.6 | 159.4 |
| TOC (mg/L) | 80.2 | 67.2 |
| SS (mg/L) | 181.3 | - |
| T-N (mg/L) | 38.5 | 40.6 |
| T-P (mg/L) | 4.5 | 3.8 |

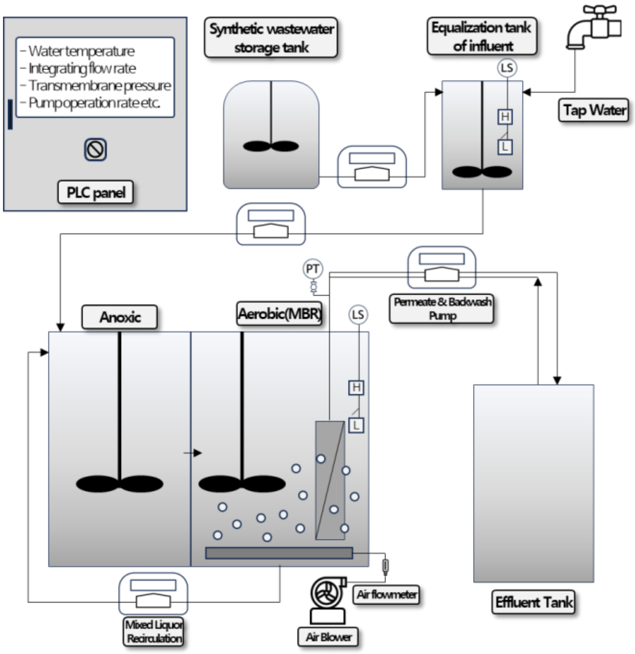


Fig. 1 A schematic diagram of the lab-scale MBR system

influent concentrations at Tancheon water reclamation center, Seoul. The concentrations of the synthetic wastewater components, including biochemical oxygen demand (BOD), chemical oxygen demand (COD), total organic carbon (TOC), suspended solids (SS), total nitrogen (T-N), and total phosphorous (T-P) are listed in Table 4.

To compare the biofouling delay capability and phosphate adsorption efficiency of the La-QQ beads, the process was operated under three different conditions. As summarized in Table 5, the control MBR lacked beads in the reactor, while the QQ-MBR and La-QQ MBR were operated with PVA-alginate beads injected at 1% of the reactor volume. The TMP and effluent quality of the experimental MBRs were analyzed.

After reaching the critical TMP and terminating the operation of the both MBR processes, an analysis of the water quality was conducted. This analysis included TOC, ammonia nitrogen ($NH_3$-N), nitrate nitrogen ($NO_3$-N), and T-P. TOC was analyzed using the Sievers M9 from GE Sievers, while $NH_3$-N and T-P were analyzed using the Hach DR 6000, along with the respective analysis kits.

## 3. Results and discussions

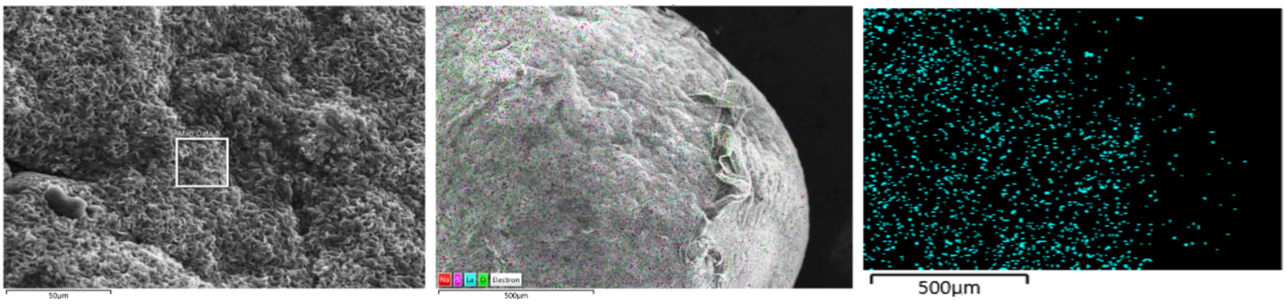
### *3.1 Evaluation of the phosphate adsorption batch test*

As a result of the bead fabrication process under the conditions mentioned in Section 2.1, beads #2 and #4 were dispersed during the distilled water washing step and completely disintegrated in the sodium sulfate solution during the second cross-linking process. However, stable beads were successfully fabricated under conditions #1 and #3. To analyze the characteristics of lanthanum doping under condition #3, surface images were captured using SEM-EDS. The surface displayed numerous pores, and elemental analysis using EDS confirmed an even distribution of lanthanum doping on the surface (Fig. 2).

The results of the batch experiments for phosphate adsorption using lanthanum-doped beads indicated that as the volume of La-QQ beads inside the reactor increased, the removal efficiency of $PO_4$-P also increased (Table 6). However, when the quantity of beads inside the reactor exceeded 1% (Cases 6-9), the concentrations of removed phosphates were similar due to saturation in adsorption (Fig. 3). The calculated unit volume removal rate of phosphate showed a decreasing trend as the volume of injected beads increased. Based on the experimental results, among the cases with excellent effluent quality, case 5 exhibited the highest unit volume removal rate. Therefore, it was concluded that Case 5 was the most efficient. This finding demonstrates that the setting bead injection volume at 1% of the MBR reactor's volume is appropriate, as per the operating conditions.

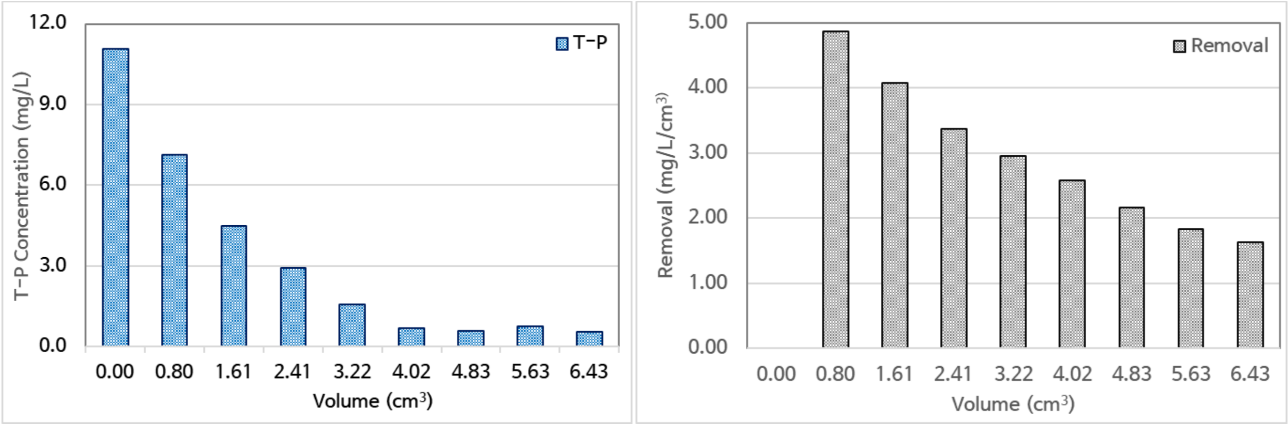
### *3.2 Evaluation of MBR performance*

To assess the efficiency of La-QQ beads in the MBR process, the system was operated under the three conditions described in Section 2.3. In the control MBR process, the $NH_3$-N influent concentration ranged from 31.0 to 43.0



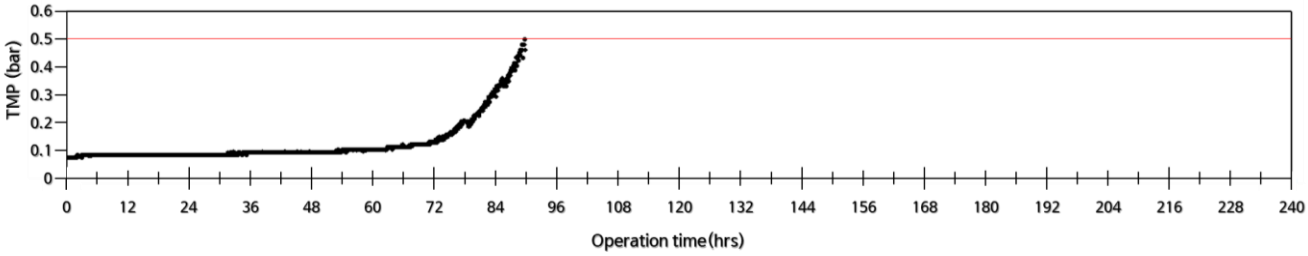
(a) electron image (b) EDS layered image (c) layered surface

Fig. 2 SEM-EDS images of the lanthanum doped beads

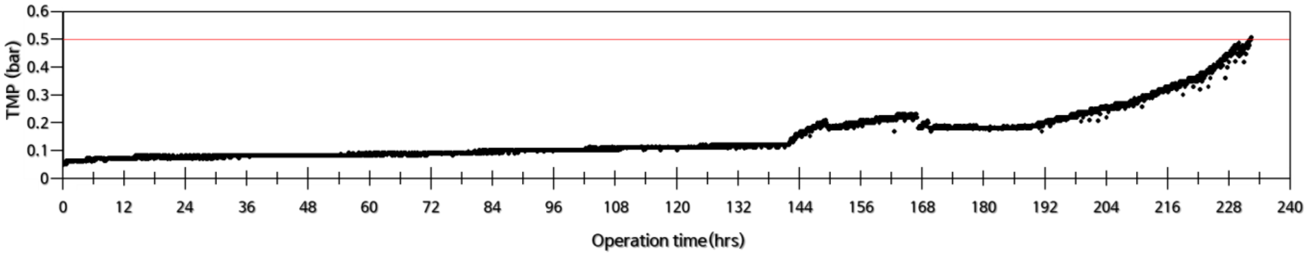


(a) T-P concentration after adsorption (b) T-P removal per unit volume of bead

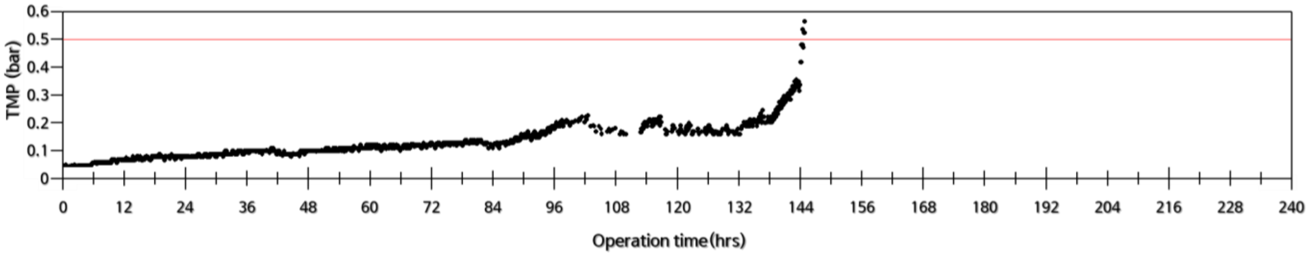
Fig. 3 Adsorption of T-P by different volumes of La-QQ beads



(a) TMP of control MBR



(b) TMP of QQ MBR



(c) TMP of La-QQ MBR

Fig. 4 TMP characteristics through the operation time under different MBR reactors

Table 5 Experimental conditions of MBR operation

| MBR | Beads conditions | Average diameter of bead (mm) | Volume ratio of beads in reactor (%) |
|---|---|---|---|
| Control MBR | No bead | - | - |
| QQ MBR | PVA-alginate by calcium | 3.0 | 1 |
| La-QQ MBR | PVA-alginate by lanthanum | 3.0 | 1 |

Table 6 Adsorbed T-P content per bead volume in different adsorption tests

| Case No. | Volume($cm^3$) | Removal($mg/L/cm^3$) |
|---|---|---|
| Case 1 | 0 | 0 |
| Case 2 | 0.804 | 4.87 |
| Case 3 | 1.608 | 4.08 |
| Case 4 | 2.413 | 3.37 |
| Case 5 | 3.217 | 2.95 |
| Case 6 | 4.021 | 2.58 |
| Case 7 | 4.825 | 2.17 |
| Case 8 | 5.630 | 1.83 |
| Case 9 | 6.434 | 1.63 |

Table 7 Analysis of influent and effluent water quality in different experimental conditions

| Samples | TOC (mg/L) | $NH_3$-N (mg/L) | T-P (mg/L) |
|---|---|---|---|
| Influent | 46.0 - 57.0 | 31.0 - 43.0 | 3.0 - 3.3 |
| Effluent of control MBR | 6.9 - 34.7 | 0.1 - 0.5 | 2.6 - 2.8 |
| Effluent of QQ MBR | 7.5 - 35.4 | 0.1 - 0.6 | 2.4 - 2.6 |
| Effluent of La-QQ MBR | 7.5 - 20.7 | 0.1 - 0.6 | 0.6 - 0.9 |

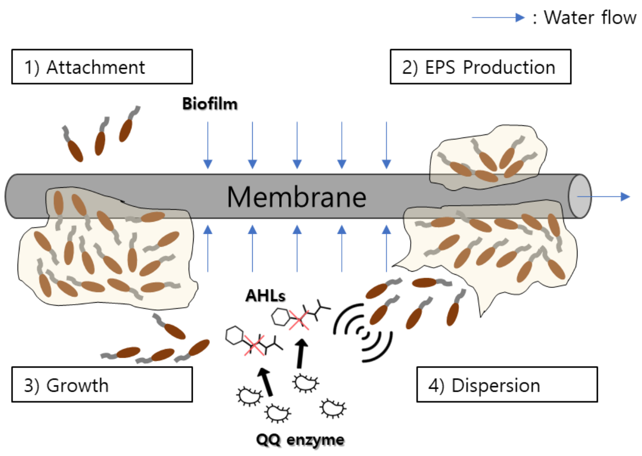


Fig. 5 Mechanism of QS and QQ on membrane biofouling

mg/L, while the effluent concentration ranged from 0.1 to 0.5 mg/L, indicating stable nitrification reactions. However, the T-P influent concentration was 3.0 - 3.3 mg/L, and the effluent concentration was 2.6 - 2.8 mg/L, indicating minimal removal through biological mechanisms.

In the QQ MBR process, the $NH_3$-N effluent concentration ranged from 0.1 to 0.6 mg/L, indicating stable nitrification reactions, but the T-P effluent concentration ranged from 2.4 to 2.6 mg/L, with only approximately 20% removal. Conversely, in the La-QQ MBR reactor, the $NH_3$-N effluent concentration remained stable at 0.1 - 0.6 mg/L, and the T-P effluent concentration was in the range of 0.6 to 0.9 mg/L, showing approximately 74% removal (Table 7). Lanthanum is initially bound to hydroxide or carbonate ions, but upon reacting with phosphate ions, it releases hydroxide and carbonate ions, subsequently adsorbing phosphate in the form of $La(H_2PO_4)_3$, leading to adsorption and removal (Koh *et al*. 2022).

Regarding the TOC removal rates under the three conditions, relatively high concentrations were discharged before the reactor stabilized. However, as the reactor stabilized over time, the removal of organic matter through aeration process ensured a stable effluent concentration.

### *3.3 Evaluation of the membrane biofouling control*

TMP profile was monitored to evaluate the delay in biofouling on the membrane due to QQ in the MBR process (Surech *et al*. 2023). In the control MBR process, where neither the physical cleaning effects of beads nor the QQ mechanism were applied, a high critical pressure was achieved within a short operating period of approximately 3.7 days.

In the La-QQ MBR, the increase in TMP was slightly delayed due to the QQ mechanism of the beads and the collision between the beads and membranes (Liu *et al*. 2021), taking approximately 6.1 days to reach critical pressure. In the QQ MBR, the increase in TMP was somewhat more delayed compared to La-QQ, taking approximately 9.8 days to reach critical pressure (Fig. 5). Increasing the time required to reach the critical pressure results in a longer cleaning cycle, which is another benefit achievable through the application of QQ beads (Pervez *et al*. 2018).

It is expected that the QQ mechanism, facilitated by QQ enzymes within the MBR reactor, would deactivate AHL signal molecules, leading to delayed membrane biofouling (Li *et al*. 2023). This analysis aimed to determine why the La-QQ MBR exhibited less effective biofouling delay compared to the QQ MBR.

## 4. Conclusions

In this study, lanthanum-doped PVA-SA beads inoculated with *BH4* were prepared and successfully used to remove phosphate and delay biofouling in a lab-scale MBR. Doping of the beads with lanthanum was highly effective in biofouling control and promoted phosphate adsorption. The unit volume removal rate of phosphate increased with the volume of the La-QQ beads. The T-P removal rate of the La-QQ MBR was 74%, which was significantly higher than that of the QQ MBR (20%) and the control MBR (14%). Beads that injected with *BH4* had been confirmed to suppress membrane biofouling in the lab-scale MBR. The time taken to reach critical pressure was approximately 9.8

days of the QQ MBR and 6.1 days of the La-QQ MBR, which was longer than 3.7 days of control MBR. A comprehensive analysis of the physical and chemical characteristics of the La-QQ and QQ beads suggests that improvements in the La-QQ beads could lead to a more stable MBR process with enhanced biofouling control and phosphate removal.

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