

Quick start-up of membrane bioreactor for treating micro-polluted surface water under low temperature

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ABSTRACT

This paper proposed an enhancement start-up strategy for accelerating the start-up of a membrane bioreactor (MBR) for treating micro-polluted surface water under low temperature. The surface water ammonia oxidizing bacteria (AOB) and nitrite oxidizing bacteria (NOB) were firstly enriched in a culture apparatus, and they were further acclimated to surface water through an MBR. Then the prepared mixed liquor obtained from the culture apparatus with natural start-up was introduced into new MBRs fed with reservoir water, and the start-up behaviors were discussed. Results indicated that the enrichment of AOB and NOB lasted for 27 days at 7.3 °C and the acclimation (at 5.9 °C) was completed within 3 days. After inoculating with the prepared mixed liquor, the start-up of the MBR was accomplished within 10 days at 9 °C with the flux of 11 L m⁻² h⁻¹ (LMH); while operated at lower initial flux (5.5 LMH) and relatively higher temperature (11 °C), it took 3 days for start-up of the MBR. However, the MBRs exhibited stable but not satisfactory organic removals under low temperature during the start-up.

Key words | drinking water treatment, low temperature, MBR, quick start-up

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INTRODUCTION

The membrane bioreactor (MBR) has attracted increasing attention due to its advantages, such as excellent solid-liquid separation, better finished water quality, reduced sludge production and automated operation (Judd 2008). As a promising technology for potable water production (Tian *et al.* 2009a), the application of MBR in drinking water treatment focuses on the removals of organics, ammonium and nitrate, though it is a relatively mature technology in wastewater treatment. Compared to the nitrate removal of MBR in some countries (Ergas & Rheinheimer 2004; McAdam & Judd 2006), MBR was employed to remove ammonium and organics in China due to its micro-polluted characteristics of surface water. Li & Chu (2003) performed an MBR for treating surface water and

found that MBR could remove nearly 60, 75 and 100% of total organic carbon (TOC), trihalomethane formation potential (THMFP) and NH₄⁺-N, respectively. However, the results reported by other researchers were not so satisfactory. Omer *et al.* (2008) only achieved the removals of approximately 25 and 30% for TOC and UV absorbance at 254 nm (UV₂₅₄), respectively. Tian *et al.* (2009a) also claimed that MBR exhibited poor removals of organics, with the average efficiencies for dissolved organic carbon (DOC), UV₂₅₄, THMFP and haloacetic acids formation potential of 21.5, 15.1, 34.1 and 24.7%, respectively. To enhance the removal efficiencies of organics, some integrative processes were developed, for example, introducing coagulant or/and adsorbent directly into the MBR (Omer

et al. 2008; Tian *et al.* 2010). Omer *et al.* (2008) dosed power activated carbon (PAC) at 2 g L⁻¹ into MBR, finding that the organic removals of hybrid PAC-MBR system were higher than that of the MBR process by at least 25%.

Although the efficiencies of MBR in organic and ammonium removal were confirmed, the start-up methods, which were of great significance, were usually different from those in previous researches (Rosenberger *et al.* 2002; Pollice *et al.* 2004; Clara *et al.* 2005; Ferraris *et al.* 2009). A standard start-up protocol to follow is still lacking. Most MBRs are operated under ambient temperature conditions, but these results could not provide effective support to MBR at low temperature (e.g. below 10 °C) (Clara *et al.* 2005; Yuan & Oleszkiewicz 2011). Thus, it is necessary to place emphasis on the study of start-up methods under low temperature. When started up using wastewater, it took the pilot-scale MBR 10 days to entirely transfer ammonium into nitrite and another 10 days was spent until nitrite was completely converted to nitrate (Rosenberger *et al.* 2002). The start-up process was accomplished within 10 days at 24 °C when MBR was initiated merely using municipal wastewater (Pollice *et al.* 2004). As reported by Guo *et al.* (2007), when inoculated with activated sludge, the durations were about 25 and 12 days for a floating media biofilter (FMB)-MBR system and a membrane adsorption bioreactor (MABR), respectively. Ferraris *et al.* (2009) carried out three start-up experiments of a pilot-scale MBR without addition of activated sludge, and reported that the durations of ammonium removal processes varied from 5 to 20 days depending on operation conditions.

Generally, methods including inoculation of domestic/municipal sludge or natural start-up (i.e. without inoculum) are adopted to initiate MBRs. Firstly, considering the safety of the finished water, the existing start-up methods are inappropriate for drinking water production due to the complexity and potential risk of the sewage components (Gil *et al.* 2011). Secondly, the period of natural start-up might be very long owing to oligotrophy at room temperature, and an even longer duration would be expected at lower temperatures. Thirdly, the AOB and NOB are ubiquitous in natural waters (Vissers *et al.* 2013), but how to enrich them is the problem needed addressing and there is limited literature about the start-up of MBR in drinking water treatment (Tian *et al.* 2009b) using diluted wastewater.

To the authors' knowledge, the approach to improve the activity of AOB and NOB in natural water and then inoculated MBR with the prepared mixed liquor has not been reported in the previous literature, especially under low temperature. The objectives of this study were two-fold: (1) to investigate the feasibility of obtaining enriched AOB and NOB from reservoir water under low temperature; (2) to estimate the start-up behaviors of MBRs inoculated with the prepared mixed liquor treating micro-polluted source water.

MATERIALS AND METHODS

Experimental set-up

The experimental set-up employed in this study was described elsewhere (Tian *et al.* 2010). A laboratory-made hollow fiber ultrafiltration (UF) membrane module (Litree Co. Ltd, Suzhou, China) was immersed inside each bioreactor (360 mL). The UF membrane was made of polyvinyl chloride (PVC), with a nominal pore size of 0.01 µm and an active membrane area of 0.02 m². The micro-polluted surface water, which was taken from a plain reservoir (the downstream of Yellow River, China), was fed through a constant level tank, and the effluent was drawn from the membrane module by a peristaltic pump (BT100-2J, Longer Pump, China). A manometer (PTP708, Tuopo Electric, China) was set between the membrane module and the peristaltic pump to monitor the trans-membrane pressure. Continuous aeration was conducted to provide sufficient dissolved oxygen (DO) for microorganisms and generate effective scouring of membrane surface, with an aeration intensity of 18 m³ m⁻² h⁻¹ (calculated by per unit area of the membrane tank). The operation of MBR was controlled by a programmable logic controller, based on a time sequence of 8 min on and 2 min off in each cycle. During the last 10 s of being off, backwash was carried out, with the flow being twice that of the permeate. No sludge was intentionally discharged from the reactor except for sampling.

Experimental protocols

To enrich the activity of autotrophic biomass (AOB and NOB), the culture process was conducted by two

sequential stages: enrichment stage and acclimation stage. During the enrichment stage, a batch reactor with the effective volume of 2 L was employed, and PAC (200 mesh, Yantai, China) at a concentration of 0.5 g L^{-1} was added into the reactor as the carrier of bacteria. The enrichment medium for surface water AOB and NOB contained the following constituents: reservoir water 1 L, NH_4Cl , 75 mg; CaCl_2 , 5 mg; NaHCO_3 , 150 mg; NaCl , 20 mg; $\text{K}_2\text{HPO}_4 \cdot 3\text{H}_2\text{O}$, 40 mg; $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, 10 mg; $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, 10 mg. The batch bioreactor was operated intermittently, based on the sequence of 22 h aeration and 2 h precipitation each day. Half of the supernatant (1 L) was drained off after precipitation and an equal volume of enrichment culture medium was added.

The acclimation stage was immediately conducted following the enrichment stage. During this stage, only NH_4Cl (75 mg L^{-1}) was added into the reservoir water and an MBR was formed by mounting a hollow fiber UF module in the reactor. The experimental set-up of MBR was similar to that described above except for the effective membrane area (0.0041 m^2) and operation mode (continuous filtration without backwashing). The permeate flux of $11 \text{ L m}^{-2} \text{ h}^{-1}$ (LMH) was carried out, corresponding to a hydraulic retention time (HRT) of 2 d.

Two parallel experiments (i.e. start-up stages I and II) were carried out to investigate the impacts of operation condition (i.e. flux in this study) and temperature on the

characteristics of MBR during the start-up. A certain volume (360 mL) of prepared mixed liquor (taken from nitrifying bacteria culture apparatus) was transferred into each reactor to start up MBRs. Unless specified, the flux of 11 LMH was applied, corresponding to an HRT of 2 h. During start-up stage II, the permeate flux was 5.5 LMH on the first several days and then it was adjusted to normal flux. It should be noted that the concentrations of $\text{NH}_4^+\text{-N}$ were maintained by the addition of NH_4Cl due to the low concentration of the reservoir water.

Analytical methods and feed water quality

The permanganate index (COD_{Mn}) was analyzed with the potassium permanganate acidic oxidation method. UV_{254} was measured using a spectrometer (T6, Puxi, China). Concentrations of $\text{NH}_4^+\text{-N}$, $\text{NO}_2^-\text{-N}$, $\text{NO}_3^-\text{-N}$ were also determined by the colorimetric methods using the spectrometer. Turbidity was analyzed by a turbidimeter (Turb550, WTW, Germany). DO was measured by the DO electrometer (pH/Oxi 340i, WTW, Germany) with a Cellox 325 probe. The pH was measured by a pH meter (Mettler Toledo, China). Temperature was determined by a mercury thermometer. Mixed liquor suspended solids (MLSS) were measured by the gravimetric method. Table 1 shows the main characteristics of the feed water.

Table 1 | The main characteristics of the feed water

Parameters	Enrichment stage	Acclimation stage	Start-up stage I	Start-up stage II
Temperature ($^{\circ}\text{C}$)	$7.3 \pm 0.8^{\text{a}}$	$5.9 \pm 0.2^{\text{c}}$	$9.0 \pm 1.1^{\text{d}}$	$11.0 \pm 0.8^{\text{e}}$
pH	$8.25 \pm 0.10^{\text{a}}$	$8.17 \pm 0.08^{\text{c}}$	$8.15 \pm 0.07^{\text{d}}$	$8.33 \pm 0.14^{\text{e}}$
Turbidity (NTU)	$5.33 \pm 1.91^{\text{b}}$	$3.37 \pm 1.01^{\text{b}}$	$3.68 \pm 1.03^{\text{d}}$	$21.9 \pm 5.6^{\text{e}}$
$\text{NH}_4^+\text{-N}$ (mg L^{-1})	$20.61 \pm 2.93^{\text{a}}$	$20.55 \pm 0.91^{\text{c}}$	$3.98 \pm 0.44^{\text{d}}$	$4.08 \pm 0.12^{\text{e}}$
$\text{NO}_2^-\text{-N}$ (mg L^{-1})	–	–	$0.012 \pm 0.001^{\text{d}}$	$0.041 \pm 0.029^{\text{e}}$
COD_{Mn} (mg L^{-1})	$3.18 \pm 0.23^{\text{b}}$	$2.85 \pm 0.42^{\text{b}}$	$3.03 \pm 0.26^{\text{d}}$	$2.45 \pm 0.13^{\text{e}}$
UV_{254} (cm^{-1})	$0.058 \pm 0.004^{\text{b}}$	$0.061 \pm 0.002^{\text{b}}$	$0.054 \pm 0.002^{\text{d}}$	$0.053 \pm 0.003^{\text{e}}$
MLSS (mg L^{-1})	$760 \pm 21^{\text{b}}$	$1,028 \pm 28^{\text{b}}$	$1,530 \pm 54^{\text{d}}$	$1,735 \pm 76^{\text{e}}$
Sludge retention time (SRT) (d)	∞	50	20	20

Note: Values are given in average \pm standard derivation. For the measurements of a, the number $n = 27$; for b, $n = 9$; for c, $n = 13$; for d, $n = 13$, for e, $n = 10$.

RESULTS AND DISCUSSION

The natural start-up performance during culture stages

Performance in the ammonium removal during culture stage

Figure 1 presents variations of nitrogen during natural start-up. It can be observed in Figure 1(a) that $\text{NH}_4^+\text{-N}$ gradually decreased since the first day of enrichment culture, indicating that there was still a small amount of AOB in reservoir water even at low-temperature (Ma *et al.* 2012; Vissers *et al.* 2013). It took approximately 2 weeks for the system to decrease $\text{NH}_4^+\text{-N}$ in the supernatant from 16.85 mg L^{-1} to less than 5 mg L^{-1} , with the removal efficiency rising from 20 to more than 80%. Further, another week was spent before the system exhibited an excellent $\text{NH}_4^+\text{-N}$ removal efficiency of approximately 95%. Finally, the $\text{NH}_4^+\text{-N}$ concentrations were maintained at a low level (less than 1 mg L^{-1}) with the removal of more than 95%, indicating the maturity of AOB.

According to the variation of $\text{NO}_2^-\text{-N}$, the enrichment stage could be further divided into four periods. It can be seen in Figure 1(a) that the concentration of $\text{NO}_2^-\text{-N}$ in the supernatant was as low as 0.01 mg L^{-1} during Period 1 because AOB in natural water was limited in activity and amount. In Period 2, the system underwent severe accumulation of $\text{NO}_2^-\text{-N}$ and the concentration increased from 0.80 to 6.95 mg L^{-1} . The $\text{NO}_2^-\text{-N}$ leveled

off (average 7.01 mg L^{-1}) in Period 3. Subsequently, $\text{NO}_2^-\text{-N}$ decreased rapidly after the 21st day (Period 4), coinciding with the maturity of the AOB 20 days later. With the concentration of $\text{NO}_2^-\text{-N}$ reduced from 5.63 to 0.75 mg L^{-1} , another week (Period 4) was spent. Finally, with the stabilization of the system, the nitrogen in the effluent was almost completely composed of nitrate and the maturity of NOB was deemed. This indicated that the process of AOB and NOB enrichment at 7.3°C lasted for 27 days.

Figure 1(b) also shows the removal efficiency of MBR during the acclimation stage. The effluent $\text{NH}_4^+\text{-N}$ decreased from 5.90 to 1.33 mg L^{-1} in the initial 3 days, with the removal efficiency increasing from 72.02 to 93.30%. Then the MBR managed to decrease effluent $\text{NH}_4^+\text{-N}$ to less than 1.00 mg L^{-1} 3 days later and the concentrations leveled off (0.28 mg L^{-1}), with an efficiency of more than 95%. It indicated that a duration of 3 days for adaptation of the MBR was spent. The factors including the lower ambient temperature (5.9°C), change of operational mode (i.e. from batch to continuous influent) and lower nutrient in reservoir water all had an adverse impact on the growth of NOB, accounting for the low removal efficiency of MBR in the initial 3 days.

Considering the period of enrichment stage (27 days), the natural start-up of MBR was accomplished within a month. The possible reasons were that the feed water was oligotrophic surface water without wastewater and the temperature in the current study was much lower than

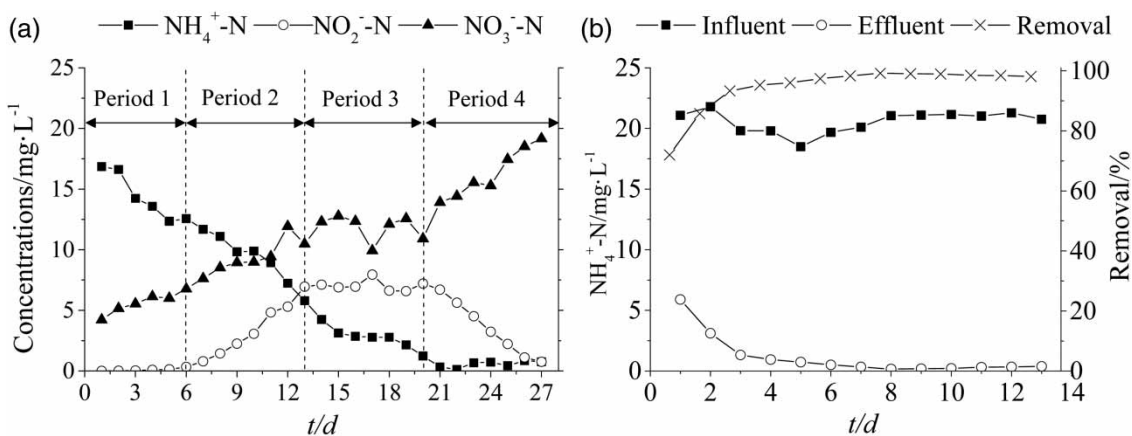


Figure 1 | Variations of nitrogen during natural start-up. (a) Concentrations of $\text{NH}_4^+\text{-N}$, $\text{NO}_2^-\text{-N}$ and $\text{NO}_3^-\text{-N}$ during enrichment stage; (b) removal of $\text{NH}_4^+\text{-N}$ during acclimation stage.

that in the previous literature (Rosenberger *et al.* 2002; Pollice *et al.* 2004).

Pollutant removal performance during acclimation stage

Table 2 lists the efficiencies of MBR in particulate and organic pollutants during the acclimation stage. The MBR exhibited excellent removal of particulate matter (97.44%), moderate COD_{Mn} removal (27.77%) but a poor removal of UV₂₅₄ (8.11%), compared with other studies (Li & Chu 2003; Omer *et al.* 2008; Tian *et al.* 2009a; Ma *et al.* 2012). A significant amount of soluble microbial product (SMP) (1.21–1.47 mg TOC L⁻¹), which were difficult to biodegrade, were produced in the long run. On the other hand, as opposed to continuous dosing in other researches (Omer *et al.* 2008; Tian *et al.* 2009a), PAC was not replenished in the current study and its adsorption capacity to small molecule weight organics was saturated.

Pollutant removal performance of MBR during start-up stages

Removals of NH₄⁺-N and NO₂⁻-N

Figure 2 illustrates the removal efficiencies of NH₄⁺-N and NO₂⁻-N by MBRs during start-up stages. In Figure 2(a), the NH₄⁺-N concentrations in the effluent of MBR had slightly increased during the first 3 days, with a peak of 1.88 mg L⁻¹. The reason might be due to the disturbance to AOB from the new operation conditions, such as lower NH₄⁺-N concentrations (reduced to 4 mg L⁻¹ from 20 mg L⁻¹) and shorter HRT (decreased to 2 h from 2 d). Figure 2(a) also shows that the effluent NH₄⁺-N of MBR decreased gradually after the 4th day, indicating that AOB and NOB had gradually acclimated to the new

environment. Moreover, the effluent NH₄⁺-N was reduced to less than 0.5 mg L⁻¹ (89.56%) on the 10th day and the MBR exhibited a pretty stable NH₄⁺-N removal (92.47%) 10 days later. On the other hand, it can be observed in Figure 2(b), the NO₂⁻-N in the effluent of MBR was observed to accumulate significantly during the first 10 days, with a peak of 0.394 mg L⁻¹ on the 5th day. However, the NO₂⁻-N began to decrease on the 6th day and gradually decreased to 0.1 mg L⁻¹ on the 10th day. Therefore, it can be inferred that the start-up of the MBR was accomplished within 10 days at 9 °C when inoculated with the prepared mixed liquor.

As shown in Figure 2(c), when initiated at 11.0 °C with a low flux (5.5 LMH), the MBR could maintain both NH₄⁺-N and NO₂⁻-N in the effluent at a low level on the first day, with the values of 0.08 and 0.113 mg L⁻¹, respectively, indicating the high activity of autotrophic bacteria in the MBR. Then the flux was adjusted to the normal value on the 2nd day, resulting in a slight increase of effluent NH₄⁺-N, with a value of 0.46 mg L⁻¹. However, the effluent NH₄⁺-N declined gradually after the 3rd day and it leveled off (0.17 mg L⁻¹), with an average removal of 95%. On the other hand, as can be observed in Figure 2(d), there was not significant accumulation of NO₂⁻-N. As the flux was adjusted to the normal value, the effluent NO₂⁻-N reached a maximum of 0.243 mg L⁻¹ on the 2nd day, but it declined after the 3rd day. Thus, the start-up process of MBR was accelerated and it took AOB and NOB 3 days for start-up of the MBR at 11 °C.

As discussed above, the relatively oligotrophic reservoir water had an adverse impact on the activity of microorganisms, but the successive addition of NH₄⁺-N created a proper environment suitable for the growth of autotrophic bacteria, so the periods of start-up at 9.0 and 11 °C were as short as 10 and 3 days, respectively.

Table 2 | Performance of particulate and organic matter during acclimation stage

Water quality indexes	Influent	Effluent	Removal (%)
Turbidity (NTU)	4.68 ± 1.87 (2.52–9.16)	0.11 ± 0.02 (0.09–0.14)	97.44 ± 0.71 (96.43–98.58)
COD _{Mn} (mg L ⁻¹)	2.93 ± 0.26 (2.55–3.22)	2.11 ± 0.33 (1.67–2.54)	27.77 ± 9.75 (12.41–40.78)
UV ₂₅₄ (cm ⁻¹)	0.063 ± 0.002 (0.060–0.064)	0.058 ± 0.006 (0.051–0.063)	8.11 ± 6.73 (1.56–15.00)

Note: Values are given in average ± standard derivation (minimum-maximum). The measurement number *n* = 9.

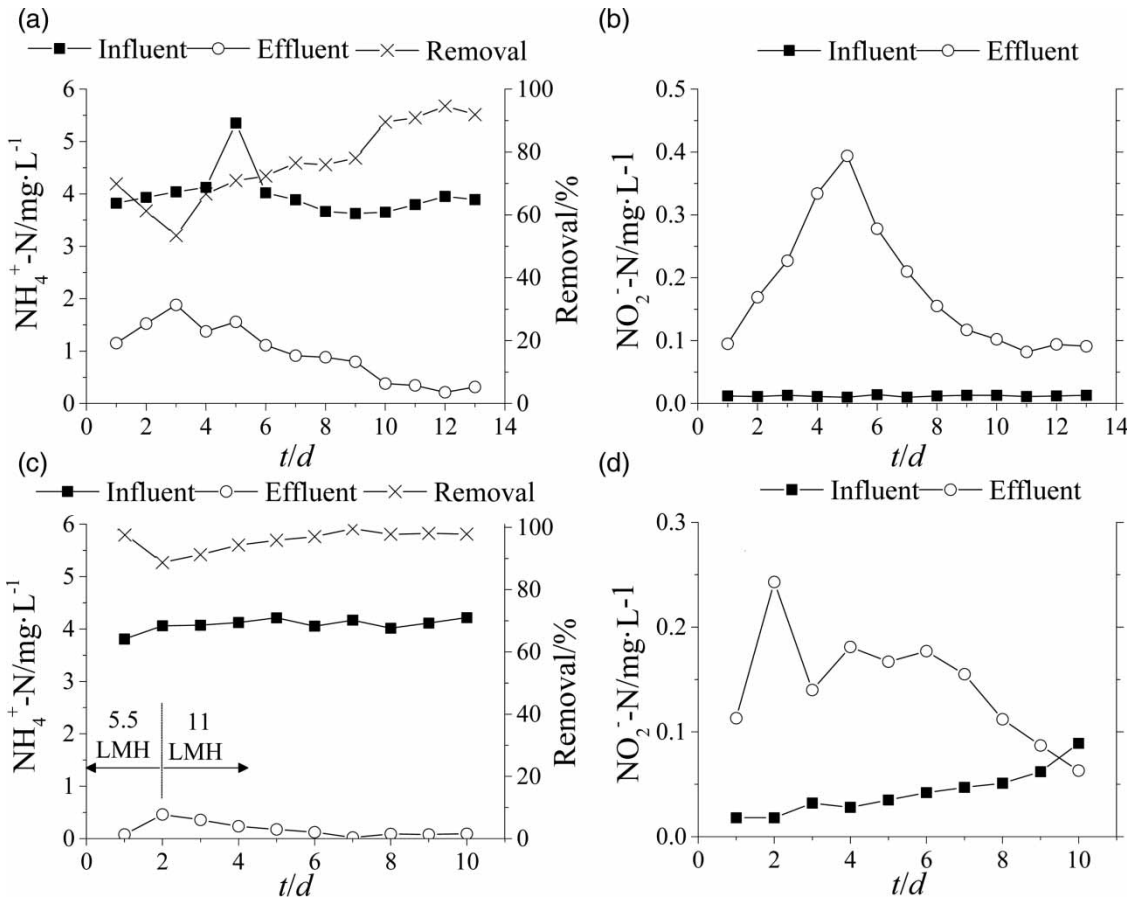


Figure 2 | Removals of $\text{NH}_4^+\text{-N}$ (a) and (c) and $\text{NO}_2^-\text{-N}$ (b) and (d) by MBR at 9.0 °C (a) and (b) and 11.0 °C (c) and (d).

The reasons for this discrepancy of different temperature were related to the higher surrounding temperature (a rise of 2 °C) and the initial reduction of permeate flux (half of the normal value). The period of start-up in the current study was shorter than the natural start-up at room temperature (17.9 ± 1.9 °C) of 20 days (Tian *et al.* 2009b), and even shorter than inoculation of wastewater sludge reported by Guo *et al.* (2007), in which about 25 days and 12 days were spent in different MBRs.

Removals of organics

The organic removal performance of MBR under 9 and 11 °C was presented in Figure 3. It is shown in Figures 3(a) and (b), during the start-up at 9 °C, that MBR could remove 19.60 and 8.89% of COD_{Mn} and UV_{254} , respectively. On the other hand, the MBR exhibited worse

removals of COD_{Mn} and UV_{254} during the start-up at 11 °C, with the average efficiencies of 12.32 and 8.67%, respectively. Compared with other studies (Li & Chu 2005; Omer *et al.* 2008; Tian *et al.* 2009a; Ma *et al.* 2012), the removals of organics by MBR were not satisfactory. It could be deemed that there was no clear indication for the maturity of heterotrophic bacteria in this study. As no sludge was discharged from the bioreactor during the experiment, not only the accumulation of inorganic but also that of organic compounds (Rosenberger *et al.* 2002) could be predicted. Thus the mixed liquor of MBR exhibited bad biodegradability and the real concentrations of organics in the mixed liquor were higher than that of the feed water. Besides, the addition of $\text{NH}_4^+\text{-N}$ enriched the activity of autotrophic bacteria at the cost of lessening that of heterotrophic bacteria.

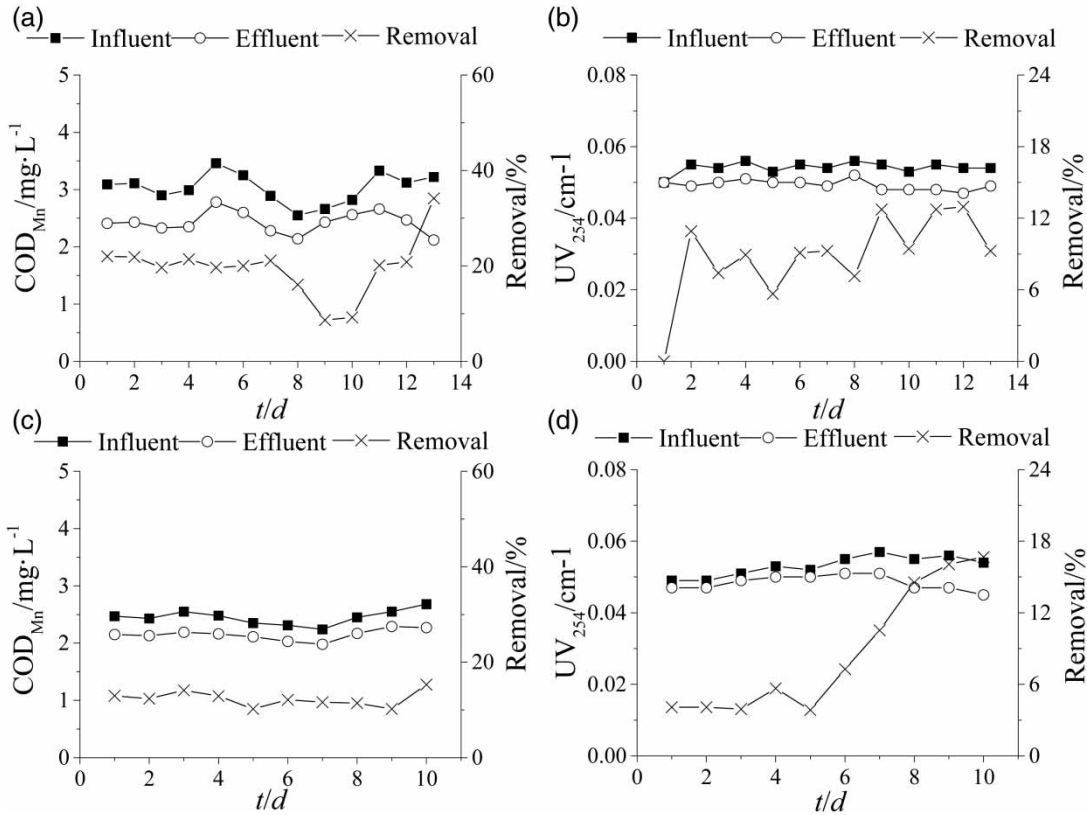


Figure 3 | Removals of COD_{Mn} (a) and (c) and UV₂₅₄ (b) and (d) by MBR at 9.0 °C (a) and (b) and 11.0 °C (c) and (d).

CONCLUSIONS

The performances of natural start-up and quick start-up of MBR for treating micro-polluted surface water were systematically investigated. The following conclusions could be drawn.

1. It was an effective approach to improve the activity of autotrophic bacteria from reservoir water using enrichment culture and the period of maturity for AOB and NOB was 27 days at 7.3 °C.
2. The acclimation (at 5.9 °C) was completed within 3 days when AOB and NOB were cultured by a MBR and the natural start-up of MBR was accomplished within a month.
3. The start-up of MBRs were accelerated significantly when they were initiated with the prepared mixed liquor. The durations were 10 days and 3 days at 9.0 °C with 11 LMH and 11.0 °C with 5.5 LMH firstly, respectively.
4. There was no clear indication for the maturity of heterotrophic bacteria during the start-up of MBR. MBRs exhibited poor organic removals under low temperature,

with the COD_{Mn} and UV₂₅₄ removal rates less than 20 and 10%, respectively.

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