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Biological removal characteristics of phenol with filtration bio-reactor

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ABSTRACT

A laboratory scale bio-reactor equipped with a filter module was examined for the separation of activated sludge, where the effluent was withdrawn by mesh filtration. The synthetic wastewater containing phenol was fed into the reactors, and the removal performance was evaluated under the following conditions: 7 or 14 d of HRT, room temperature (ca. 20°C), synthetic wastewater containing 5,000–20,000 mg/L of phenol, 262 mg/L of NH_4-N, 43.7 mg/L of PO_4-P, maximum phenol loading rate: 1.43 kg/m^3/d, feeding the synthetic wastewater for 5 or 10 h (Run-1) or feeding at once (Run-2), and filtration once a day. The reactors maintained high concentrations of MLSS (10,000–15,000 mg/L), and the filtration was carried out stably in a short time. When the synthetic wastewater containing 5,000 mg/L of phenol was fed into the reactor at 7 d of HRT (0.7 kg/m^3/d of phenol loading rate), in the operation of Run-1, the removal rates for phenol, NH_4-N, and D-TN were above 99.9, 98, and 96%, respectively. Similar results were also obtained in the operation of Run-2. The microbial activity for phenol decomposition was influenced slightly by phenol-MLSS loading rate, but it was significantly affected by the initial phenol concentration in Run-2. The sludge properties such as floc size and content of exocellular polymer affected the filtration performance of this system.

Keywords: Filtration bioreactor; Phenol; Nitrogen removal; ECP

1. Introduction

The performance of biological wastewater treatment process is influenced significantly by the inflow of toxic substances [1–3], particularly the phenolic compounds, which are typical toxic substances that inhibit the activity of microbes. Since conventional aerobic biological treatments are difficult to be applied for the removal of wastewater containing toxic and/or refractory organic compounds, the physicochemical and/or chemical oxidation processes are widely employed instead, for example, activated carbon adsorption [4], solvent extraction [5], and chemical oxidation [6]. In case of employing biological processes to this purpose, the following advantages may be expected: reduction of chemicals, less production of sludge, easy operation, and cost-effectiveness [7–11].

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When toxic substances are treated by a biological process, it is essential to acclimate and to enrich the biomass for the removal of substances. The enhanced and satisfactory operations can be anticipated under conditions that a high concentration of biomass is retained and the influent is properly loaded against the response of microbial growing even in the presence of inhibitory compounds [12,13]. In spite of phenolic toxicity to activated sludge, the acclimated biomass can effectively degrade phenol [9].

A membrane bio-reactor (MBR) can retain high concentration of biomass, and some researchers examined MBR for the removal of phenolic compounds [14,15]. The advanced performances of a MBR system are caused by the following factors: complete rejection of washout of the acclimated biomass, retention of high concentration of biomass, and very long sludge retention time.

A mesh filtration device can also effectively separate and enrich activated sludge without the help of a suction pump [16]. A mesh filtration bio-reactor, which is equipped with a mesh module instead of a membrane module for sludge separation, can effectively retain high concentrations of MLSS, and has high potential for an alternative bio-reactor [17,18]. In a mesh filtration bio-reactor system, since the sludge separation is conducted by the sludge layer formed at the mesh surface, it is required to maintain an appropriate sludge layer for effective filtration [18].

In this work, the mesh filtration bio-reactor was operated as a sequencing batch reactor and was applied for the removal of phenol. The properties of the system were examined from the following viewpoints: sludge separation properties, the MLSS concentration, the removal performance for phenol and nitrogen including the loading rates and their concentrations in the feeding solution, and effluent quality. In addition, the contents of exocellular polymers (ECPs) of the sludge were examined as one of the key factors on the filtration properties.

2. Materials and methods

2.1. Reactor system

The treatment system was made of a cylindrical acryl tank (19 cm i.d × 50 cm height; working volume: 7 L) equipped with a mesh filter module at the bottom of the reactor. Nylon mesh was of 100 μm opening size (opening ratio: 44%) and 518 cm^2 of effective mesh area. In our pervious works, it was found that the mesh was appropriate for sludge separation [17,18]. A schematic diagram of this system is shown in Fig. 1.

Other apparatuses such as a feeding pump and a recycling pump to return the initial filtrate into the reactor were employed in this work. Two aerators were used in the reactor; One was placed at the bottom of the reactor to supply oxygen for the microbes, and the other was located below the filter module to remove the accumulated sludge layer at the mesh surface of filter module. The filtration was carried out only by the hydraulic pressure difference between water level in the reactor and effluent port. The initial water head was adjusted to 800 mm, and it was decreased a little with the progress of filtration. Since the initial filtrate contained a high concentration of SS, it was returned into the reactor and the filtrate after a few minutes of operation was collected as effluent. The period required to obtain the effluent was defined as the filtration time.

2.1.1. Wastewater treatment

The activated sludge obtained from the domestic wastewater treatment facility was fed into the rectors as a seeding biomass. The synthetic wastewater was prepared with phenol and some inorganic salts (NH_4-N: 262 mg/L; PO_4-P: 43.7 mg/L, alkalinity: 238 mg/L; $CaCl_2{\cdot}2H_2O$: 10 mg/L; and $MgSO_4{\cdot}7H_2O$: 30 mg/L), and it was fed daily into the reactors by the following procedures: feeding continuously for 5 or 10 h (Run-1), and feeding at once (Run-2). The effluent was withdrawn once a day by the mesh filter module.

The operational conditions for two reactors are summarized in Table 1. The periods from Mode-A to Mode-D were the acclimation period and the phenol loading rates were increased gradually. In the period of Mode-G, the synthetic wastewater was not fed. Only Mode-H was operated at HRT = 14 d and the other periods were operated at HRT = 7 d. The experiments were conducted at room temperature (ca. 20 ℃). The concentrations of phenol were varied in the range of 5,000–20,000 mg/L, and the volumetric phenol loading rates were in the range of 0.714–1.43 $kg/m^3/d$.

As presented in a previous work [18], the batch-type operation was conducted in the sequence of following procedures: (1) the attached sludge layer on the mesh filter was removed by aeration cleaning, (2) the initial filtrate just after starting the filtration (for ca. 3 min: ca. 1 L) was returned to the reactor, because the initial effluent contains high concentration of SS, (3) after that the filtrate was collected as an effluent (the biomass was separated effectively by sludge layer formed at the mesh surface), (4) the attached sludge layer formed during the filtration practice was removed again by aeration cleaning, and (5) the excess

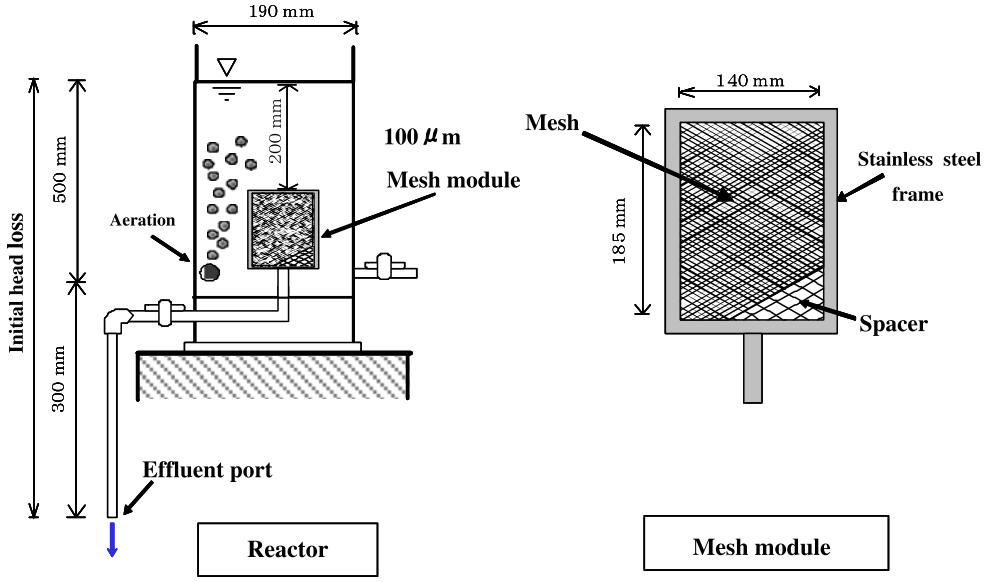

Fig. 1. Schematic diagram of the mesh filtration bio-reactor.

Table 1
Operational conditions

Mode	period (d)	Synthetic wastewater			Feeding method			
		Phenol conc. (mg/L)	Volume (L/d)	Volumetric loading (kg/m^3/d)	Run-1	Run-2	HRT (d)	DO (mg/L)
A	0–20	200	0.5	0.014	Start-up period (Step feeding)(1)		14	4.0–6.5
B	21–34	2,000	0.5	0.142				
C	35–49	4,000	0.5	0.286				
D	50–62	8,000	0.5	0.571				
E	63–130	10,000	0.5	0.714	Continuous(2)	Slug dose(3)	14	
F1	131–171	20,000	0.5	1.428	Continuous(2)	Slug dose		2.0
G	172–179	–	–	–	–	–	–	2.0
F2	180–223	20,000	0.5	1.43	Continuous(2)	Slug dose	14	2.0
H	224–292	5,000	1	0.714	Continuous(4)	Slug dose	7	2.0

Note: (1) Feeding over several hours; (2) continuous feeding for 5 h; (3) feeding at once; (4) continuous feeding for 10 h. No substrate was fed during the G period.

sludge was fed and the reactor was aerated continuously. Phenol was analyzed by the distillation–photometric method [19] and the other water quality items were determined by US Standard Methods [20].

2.2. Oxygen uptake rate

The parameter of oxygen uptake rate (OUR) was measured to evaluate the effect of the phenol feeding method on the activities of microbes. Among the measuring methods for OUR [21–23], the following method was applied: the decrease of DO in a closed vessel was periodically measured on the basis of a respirometer by Warburg apparatus, where the oxygen consumption was monitored with a BOD sensor (BOD Sensor System, ACTAC, Japan) at 20°C. The biomasses obtained from the mesh filtration bio-reactors were adjusted in the rage of 500–1,000 mg/L, and the phenol concentration was varied. The oxygen uptake was measured at every one hour for 1 d, and OUR was

calculated by the slope of oxygen uptake against time. In addition, OURs were measured by other substrates such as skim milk (BOD = 200 mg/L), glucose (1,000 mg/L), and NH_4Cl (1,000 mg/L).

2.3. ECP and filtration properties

ECPs were extracted from the biomasses in two reactors (Run-1 and Run-2) by the method described in a previous work [18]. The contents of carbohydrates and proteins in the ECPs were analyzed by the phenol–sulfuric acid method [19] and the Lowry method [24], respectively.

3. Results and discussion

3.1. Effect of solids on the filtration characteristics

As the profiles of MLSS are shown in Fig. 2, the MLSS in the reactors increased during the acclimation period (Mode-A to -D), and the reactor retained more than 10,000 mg/L of MLSS. The filtration times were also presented in Fig. 2. Although long filtration time was observed frequently, a few hours of the filtration time may be allowed taking into consideration, the overall operation time of the sequencing batch operation (Run-2).

However, Fig. 2 shows the decrease of MLSS, and the filtration time increase during the periods. The decrease of MLSS was caused by the deflocculation of sludge; since the dispersed sludge flocs were produced, the sludge cake layer was not formed effectively, the filtration time increased, and the high concentration of SS passed through the mesh filter, as shown in Fig. 3. In the period of Mode-E, -F1, and -F2, a large amount of fine flocs was observed, and the phenol removal performances were somewhat reduced as described later. The filtration times of Run-2 were generally longer than those of Run-1, and the feeding method may affect the properties of sludge.

Since the phenomena were not resolved naturally, the fine flocs were removed in the final stage of the Mode-F2, when the settleable sludge was collected and fed back to the reactors. In the case of Mode-H, filtration was carried out stably in a short period of time (ca. 3 min for Run-1 and ca. 80 min for Run-2). Even during the Mode-H, however, the effluents contained relatively high SS concentration: average concentrations of SS were 33 mg/L for Run-1 and 74 mg/L for Run-2, respectively One of the reasons

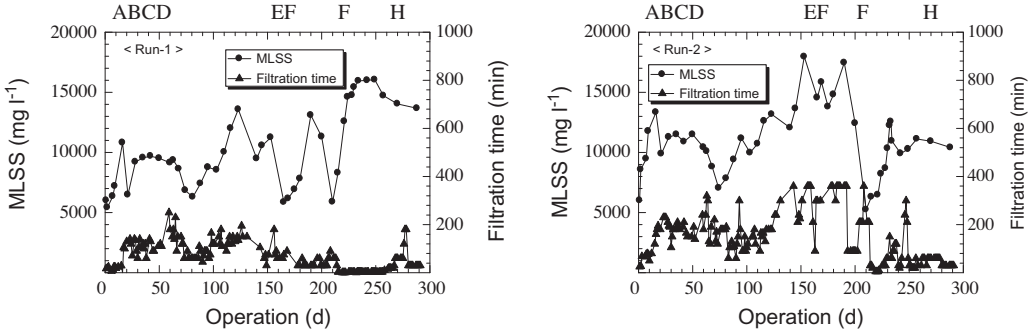

Fig. 2. Profiles of MLSS and filtration time.

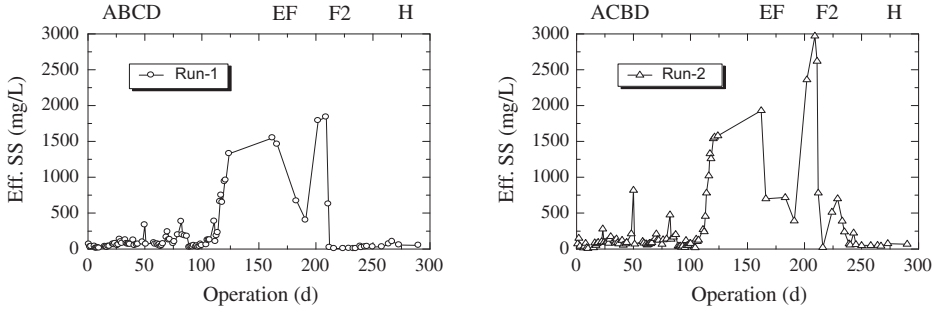

Fig. 3. Profiles of SS of the effluents.

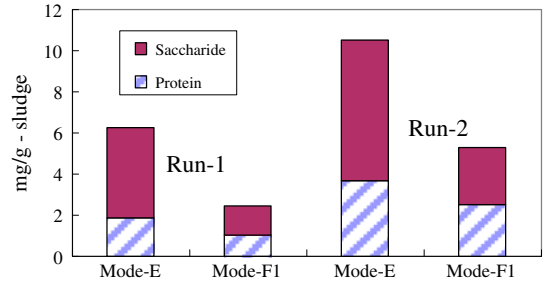

Fig. 4. ECP contents of the sludges (Mode-F1: clogging period).

may be caused by short time of returning the initial effluent. The pH conditions (6.9–7.8) in the reactors may affect the filtration properties. In our previous works [17,18,25], it was found that better filtration performance was obtained under acidic condition than under neutral and alkaline conditions.

3.2. Relationship between ECP and filtration properties

The ECP contents of sludges in the reactors were measured in Mode-E and Mode-F1. The clogging of mesh filter was observed in the latter period. The ECP contents are shown in Fig. 4.

In the period of Mode-F1, the ECP contents were significantly lower than those in the period of Mode-E, and corresponded to those obtained under neutral pH and alkaline conditions in a previous work [18], where the clogging of mesh was observed. In addition, it was pointed out that even ECP contents in Mode-E were lower than those under the acidic conditions in a previous work [11]. It was suggested that the sludge containing high saccharide content in ECPs filtrated in a short time [18]. Therefore, the production of fine flocs and the clogging of mesh may have been caused by the low content of ECP in the sludge. The ECP may play an important role on the adhesion of the sludge flocs, although the controlling factors for ECPs content in the sludge have not presently clarified.

3.3. Removal properties of phenol

The concentration variations of effluent phenol and removal efficiencies are presented in Fig. 5. Relatively high concentrations of phenol in the effluents were observed in Mode-F1 and Mode-F2; however, the removal efficiencies were more than 98%, where the phenol concentration of feed solution was 20,000 mg/L, and the volumetric phenol loading rate was very high at 1.43 kg/m^3/d. The maximum phenol loading rates including other published results [9–11] are summarized in Table 2. The volumetric loading rates were more than two times higher than the others, and F/M ratios (g-Phenol/kg-MLSS/d) were also higher at 1.4–2.4 times. These results indicated that this system could effectively acclimate and enrich the microbes assimilating phenol.

The fine flocs were produced in the periods of Mode-F1 and Mode-F2, and the filtration performance decreased as mentioned previously. This corresponded to very high loading rate, and therefore, in the period of Mode-H, the reactors were operated once more at 0.714 kg/m^3/d of the loading rate, where 1 L of the feed solution containing 5,000 mg/L of phenol was fed and HRT was 7 d. The effluent phenol concentrations were stably maintained lower than 0.1 mg/L in the case of Run-1. In the case of Run-2, the phenol concentrations were high in the initial period but decreased rapidly to be kept at a concentration lower than 0.1 mg/L.

3.4. Phenol removal with different feeding type

Continuous feeding (Run-1) and slug feeding (Run-2) methods were examined in this work. The variation of the phenol concentrations in the reactors were monitored in Mode-E and the results are shown in Fig. 6.

In the case of Run-1, where the phenol solution was fed continuously for 10 h, the phenol concentrations in the reactor increased only to ca. 8 mg/L. On

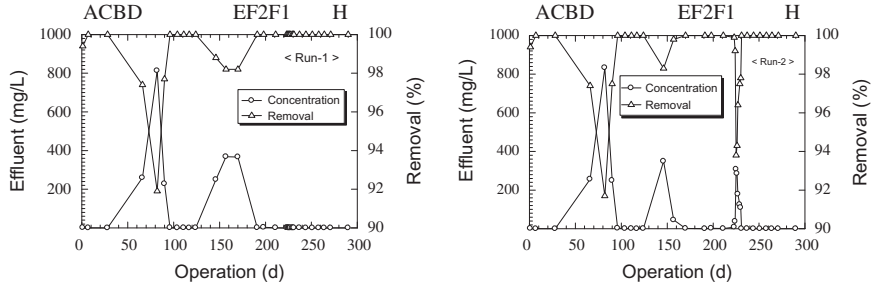

Fig. 5. Profiles of effluent concentration and removal of phenol.

Table 2
Maximum phenol loading rates

	g-Phenol/kg-MLSS/d	kg-Phenol/m^3/d
Run-1	0.242	1.428
Run-2	0.272	1.429
Okada [9]	0.175	0.700
Watanabe [10]	0.167	0.333
Yamagishi [11]	0.100	0.500

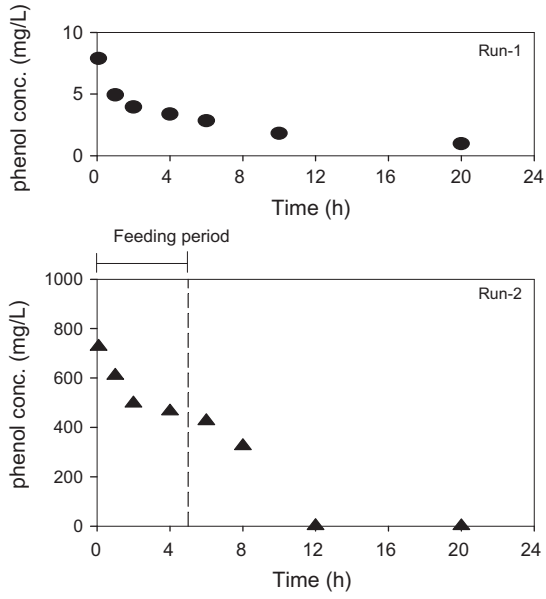

Fig. 6. Effects of phenol feeding method on OUR values.

the other hand, in the case of Run-2, the phenol concentration in the reactor was 727 mg/L, just after the feeding, and decreased gradually to a lower concentration of less than 1 mg/L for 12 h. Okada et al. [9] indicated the importance of feeding method of phenol from different types of operation, in which SBR operation would be preferable to build up and maintain the activity of microbes even with the same daily loading of phenol. However, very high concentration of phenol may offer some stress to the microbes, taking into consideration the results for very high phenol loading rate (Mode-F1 and -F2).

3.5. Oxygen uptake activity

The effects of phenol concentration on the activity of the microbes in the reactors were examined by OUR, which is simply employed for the evaluation of the activities of heterotrophs [25]. The effects of phenol concentration on OUR were examined with the following sludges: Sludge-1 was obtained from the domestic wastewater treatment facility and Sludge-2 from the mesh filtration bio-reactor in Run-1 (Mode-F2; the flocculated sludge). The results are summarized in Fig. 7. In the case of Sludge-1, although OUR increased slightly at very low phenol concentration, significant oxygen uptake was not observed. The OUR of the Sludge-2 increased significantly at more than 0.1 mg/L of phenol concentrations, and the results indicated that the sludge assimilated phenol.

The effects of substrate on the OUR were also examined with Sludge-1 and Sludge-2, and the results are summarized in Table 3. Sludge-1 indicated the highest OUR value for skim milk and the lowest values for NH_4Cl and phenol. In the case of Sludge-2, the highest OUR value was observed for phenol and the OUR value for NH_4Cl was also higher than that of Sludge-1. The results shown in Fig. 7 and Table 3

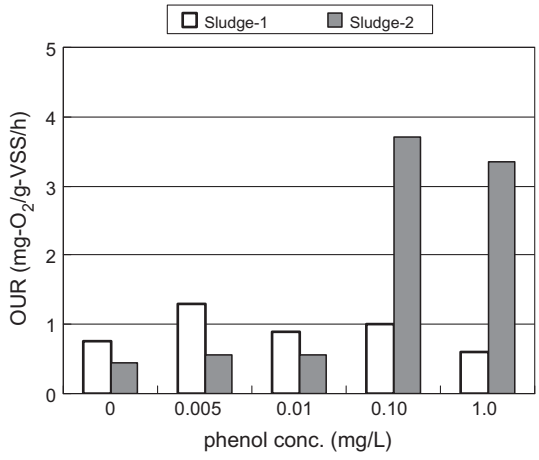

Fig. 7. Effects of phenol concentration on OUR.
Notes: Sludge-1: obtained from domestic wastewater treatment facility (extended aeration). Sludge-2: obtained from the reactor in Run-1 (Mode-F2: flocculated sludge).

Table 3
Effects of substrate on OUR (Mode-H)

	OUR (mg-O_2/g-VSS/h)	
Substrate (conc.)	Sludge-1	Sludge-2
Blank	0.60	0.59
NH_4Cl (1 g/L)	0.36	0.95
Skim milk (BOD: 200 mg/L)	6.05	0.36
Glucose (1 g/L)	0.82	1.14
Phenol (1 g/L)	0.36	3.77

Note: Sludge-1: obtained from the domestic wastewater treatment facility. Sludge-2: obtained from the reactor of Run-1.

Table 4
Effluent qualities (Mode-H: 247–275 d)

Item	Influent (mg/L)	Run-1 (0.7 kg/m^3/d)		Run-2 (0.7 kg/m^3/d)	
		Range	Average	Range	Average
D-TN	–	1.2–26.3	9.1	1.1–45.7	22.5
NH_4-N	262	0.1–20.6	5.3	0.5–28.4	9.4
NO_3-N	–	0–3.1	0.9	0–2.6	0.6
Phenol	5,000	0.03–0.07	0.05	0.05–0.09	0.07
Colority	–	23–30	27	33–102	40
Filtration (min)		3	3	20–210	76

Note: Unit: mg/L except colority expressed as mg Pt/L.

indicated significantly that the reactor retained the biomass having high activities for phenol decomposition and for nitrification.

3.6. Filtrate water qualities

The average concentrations of nitrogen, phenol, and the colority of effluents in Mode-H are summarized in Table 4, where the volumetric phenol loading rate was 0.714 kg/m^3/d, HRT was 7 d, and the concentrations of MLSS were 14,000–16,000 mg/L for Run-1 and ca. 10,000 mg/L for Run-2. The phenol concentrations of effluent were less than 0.1 mg/L for both the cases. High colority was observed in the case of Run-2, and it was also observed in the acclimation period from Mode-A to Mode-D. This may be caused by self-oxidation of the sludge which cannot assimilate phenol. Similar results were commonly observed in the acclimation period for other refractory organic compounds with a mesh bio-reactor.

Although the feeding solution contained 262 mg-N/L of NH_4, the average NH_4-N concentrations in the effluents were 5.3 mg-N/L for Run-1 and 9.4 mg-N/L for Run-2, and nitrification was almost completely conducted. Considering the fact that the biomass indicated relatively higher OUR value for NH_4Cl (Table 3), it is obvious that the reactors also retained and enriched nitrifying microbes effectively. In addition, the NO_3-N concentrations were kept at lower than 1 mg/L, and therefore denitrification was also completely performed. As the results, the overall nitrogen removals were in the range of 82.6–99.6%, where the average removal rates were 96.5% for Run-1 and 91.4% for Run-2. The results indicated that the reactors were effective for simultaneous removal of phenol and nitrogen.

4. Conclusions

Bio-reactor equipped with a mesh filter was applied for phenol removal and the effective removal performance was obtained. The results obtained in this work are summarized as follows:

- High concentrations of MLSS (10,000–15,000 mg/L) were effectively retained in this system, and the filtration was carried out stably in a short time.
- The effluent phenol concentration lower than 1 mg/L was maintained when 5,000 mg/L of phenol was fed with 7 d of HRT.
- When the phenol loading rate was 1.43 kg/m^3/d, phenol was removal more than 98%, although the sludge was deflocculated and the solid separation performance was reduced.
- When phenol was fed at high loading rate, it is suggested that stable treatment performance is obtained by continuous feeding method.
- The filtration performance was influenced by the floc size of the sludge, and the ECP content of the sludge affected the floc size and the filtration performance.

Considering that this system is a simple set-up and can be operated easily, it is not only a useful bio-reactor system but also may be of use as a reactor for preliminary experiments for the removal of toxic and refractory organic compounds prior to the application of MBR device.

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