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Characteristics of aerobic granules treating phenol and ammonium at different cycle time and up flow liquid velocity

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ABSTRACT

Synthetic wastewater containing phenol (400 mg L^{-1}), thiocyanate (SCN^-) (100 mg L^{-1}) and ammonia-nitrogen (NH_4^+-N) (100 mg L^{-1}) was treated in the three aerobic granular reactors at different cycle time of 6 h (R1), 12 h (R2) and 24 h (R3) with organic loading rate (OLR) of 2.03, 1.02, and 0.51 kg COD m^{-3} day^{-1}, respectively. Reactor performance was analyzed in terms of granule characteristics and organics and nitrogen removals. Granule size, biomass concentration and extracellular polymeric substances (EPS) in sludge were inversely related to cycle time. In R1, mean biomass size and volatile suspended solids (VSS) were 1334.24 ± 30.56 μm and 4.31 ± 0.40 g L^{-1}, as compared to 97.93 ± 4.21 μm size and 2.28 ± 0.13 g L^{-1} VSS in R3. Sludge EPS in R1 was two-fold more than R3. The ratio of protein (PN) to polysaccharide (PS) content in sludge increased with an increase in loading. Pollutant removal performance was independent of cycle time with 94–96% removal of COD (chemical oxygen demand) and 99% removals of phenol, SCN^-, and NH_4^+-N. In reactors R1 and R3, nitrification was complete to nitrate, and in R2, 40% of influent NH_4^+-N was converted to nitrite. Up flow liquid velocity of 2.5 m h^{-1} was appropriate for better granulation and can serve as a selection pressure for granulation.

1. Introduction

The economic and satisfactory treatment of industrial wastewaters like from oil refineries, coal gasification, coking plants, petrochemicals, and pharmaceutical etc., is a great challenge since they are comprising of complex matter consists of several organic compounds (like aromatic compounds), inorganic compounds and ammonia (Kim and Kim, 2003; Ramos et al., 2016b). Nonetheless, the aromatic compounds can cause inhibition to the biological processes (Al Khalid and El Naas, 2012). Phenol (concentration 110–487 mg L^{-1}) is accompanied with other toxic and inhibitory compounds like thiocyanate (SCN^-) and ammonia-nitrogen (NH_4^+-N) in wastewaters from coal gasification, synthetic fuel processing, coal liquefaction etc. (Li et al., 2011; Vázquez et al., 2006; Zheng and Li, 2009). When phenol and thiocyanate are present with ammonia, these compounds inhibit nitrifying bacteria, hence require to be removed prior nitrification (Jeong and Chung, 2006; Kim et al., 2008b). In wastewater treatment, removal of ammonia by nitrification step is always considered as the rate limiting step due to the very slow growth rate of nitrifying bacteria and are very susceptible to several factors like inhibition by aromatic compounds (Ramos et al., 2016a).

Aerobic granule based biological treatment systems serve as an alternative approach for treating the complex industrial wastewaters (Gao et al., 2011a). Aerobic granular reactor (AGR) can retain abundant biomass by self-aggregation of microorganisms without any support/carrier (Beun et al., 1999; Liu and Tay, 2007b; van den Akker et al., 2015). Aerobic granulation has a high potency for simultaneous removal of both organic and ammonium pollutants in a cost-effective manner (Singh and Srivastava, 2011). Very few works of literature are available for the simultaneous removal of phenol with ammonia nitrogen by AGR (Liu et al., 2005; Ramos et al., 2016a, b). The restriction in the efficient operation of granules for nitrification can be overcome by adopting an adequate approach to enhance nitrifying granulation for efficient ammonia conversion (Wu et al., 2017). In AGR nitrifying bacteria can stay at the inner layer of granules and are protected from toxic compounds present in bulk solution by diffusion barrier and simultaneous organic and nitrogen removals may be achieved (Liu et al., 2005; Yang et al., 2005). Corsino et al. (2016) and Jemaat et al. (2014) reported partial ammonia oxidation to nitrite in the presence of high salinity and para-nitrophenol. The performance of AGR and granules characteristics depend on several operating parameters like hydraulic retention time (HRT), wastewater composition, settling time, airflow velocity etc. (Beun et al., 1999; Liu and Tay, 2015; Tay et al., 2001). AGR is largely operated as sequencing batch reactor (SBR) with several phases like the filling of feed, reaction, settling and decanting of

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supernatant in a cycle. In any SBR, the cycle time is interrelated to hydraulic retention time (HRT) and substrate loading of the reactor and higher cycle time gives longer HRT and lower substrate loading. When cycle time i.e. HRT is too short, microbial growth can be hindered by insufficient reaction time; on the other hand, when cycle time is too high, hydrolysis of biomass may occur by starvation of biomass, resulting in a negative impact on biomass aggregation (Liu and Tay, 2015). To the best of the knowledge of authors, no study addressed the effect of cycle time, loading, and up flow liquid velocity (ULV) on nitrification efficiency in AGR in the presence of toxic compounds like phenol and thiocyanate. Previous studies on parameter optimization in AGR were mainly carried out with mainly acetate, sucrose as feed (Liu and Tay, 2007a, b; Wang et al., 2005).

The present study aims to investigate the effect of cycle time and loading on development and characteristics of granules and performance of AGR in removing toxic pollutants like phenol, thiocyanate and to achieve nitrification. The feasibility and performance of AGR are also evaluated under the effect of ULV, a new selection pressure.

2. Materials and methods

2.1. Experimental set-up

The present study was carried out in three laboratory scale reactors operated in sequential batch mode. Each reactor had a working volume of 6 L. The working height and inner diameter (ID) of reactors were 212 and 6 cm, respectively (H/D ratio of 35). Aeration was provided by an oil-free compressor at a rate of 2 L min^{-1} by air stones kept at the bottom in all the three reactors. The influent synthetic wastewater was introduced from the bottom of the reactors with the help of peristaltic pumps. The reactors were maintained at room temperatures (25–30 °C).

2.2. Characteristics of seed

The inoculum was collected from activated sludge unit of wastewater treatment plant of Indian Oil Corporation Limited (IOCL), Noonmati, Guwahati, Assam. Total suspended solids (TSS) and volatile suspended solids (VSS) in IOCL sludge were 2.42 ± 0.16 and 1.72 ± 0.15 g L^{-1}, respectively. The particle size of sludge was of 32.78 ± 0.01 μm. The settling velocity and SVI_{30} were 2.63 m h^{-1} and 50.13 mL $gTSS^{-1}$, respectively. In all three reactors, 3 L sludge was used for working volume of 6 L.

2.3. Feed characteristics

The influent synthetic wastewater consisted of phenol of 400 mg L^{-1}; ammonia nitrogen (NH_4^+–N as NH_4Cl) of 100 mg $L^{-1}$and thiocyanate (SCN^- as KSCN) of 100 mg L^{-1}. Feed pH was maintained between 7.5 and 8 by using sodium hydrogen carbonate and phosphate buffer (using KH_2PO_4 72.3 g L^{-1} and K_2HPO_4 104.5 g L^{-1}). This phosphate buffer also worked as a phosphorus source for microorganisms. Phosphate buffer of 1 mL L^{-1} and trace metals solution of 1 mL L^{-1} was added in the synthetic feed in three reactors. The stock trace metal composition was taken from previous literature (Sahariah and Chakraborty, 2011). The composition of stock trace metal solution was: $MgSO_4.7H_2O$: 10,000 mg L^{-1}, $CaCl_2.2H_2O$:10,000 mg L^{-1}, $FeCl_3.6H_2O$: 5000 mg L^{-1}, $CuCl_2$: 1000 mg L^{-1}, $ZnCl_2$: 1000 mg L^{-1}, $NiCl_2.6H_2O$: 500 mg L^{-1}, $CoCl_2$: 500 mg L^{-1}.

2.4. Operational strategy

The operational schedule is given in Table 1 and the reactor picture is given in Fig. 1. The reactors were operated in the sequential batch mode with a volume exchange ratio of 50%. i.e. 50% of reactor working volume was decanted in each cycle and the similar amount of fresh feed was added to the reactor. Settling time was kept constant for 5 min in three reactors throughout the study, except for the initial 15 days, when it was 15 min to prevent severe washout of biomass from the reactors. Cycle time was varied as 6 h in R1, 12 h in R2 and 24 h in R3. HRT (day) was calculated using equation (1) and HRT values are given in Table 1.

$$\text{HRT (day)} = \frac{\text{Reactor volume (L)}}{\text{Volume decanted per cycle (L)} \times \text{No. of cycles per day}} \quad (1)$$

ULV was kept at the rate of 2.0 m h^{-1} in all three reactors during the cycle time study and ULVs of 2.5 and 3.0 m h^{-1} were maintained in reactor R1 for examining ULV effect on granular sludge characteristics and performance. Equation (2) was used for calculating ULV and values of ULVs are given in Table 1 with the operation time.

$$\text{ULV(m h}^{-1}) = \frac{\text{Flow rate (m}^3\text{/cycle)}}{\text{Feeding time per cycle (h)} \times \text{Cross sectional area of reactor (m}^2)} \quad (2)$$

During acclimatization period, concentrations of phenol, thiocyanate, and ammonia were increased gradually in the feed. In order to supply suitable carbon source during acclimatization, sodium acetate was added initially in the feed (1000 mg L^{-1}). With the progress of acclimatization, concentrations of feed pollutants were increased and concentration of sodium acetate was gradually decreased in the feed. After 45 days of acclimatization, there was no sodium acetate in the reactors and phenol, ammonia nitrogen and thiocyanate concentrations were increased to 400, 100 and 100 mg L^{-1}, respectively. Then reactors were operated for another 27 days with same feed and same operating condition and steady state data was collected.

2.5. Analytical methods

The granule size was measured by a laser particle size analyser (Mastersizer 2000; Malvern Instruments) and occasionally by field emission scanning electron microscope (FESEM) (Sigma, Zeiss). The sample was analyzed three times by laser particle size analyser and average value was produced. The particle size also produced the D50 value, which is the average or median diameter in μm. D50 value represents the 50 percentile of size distribution (Zhang et al., 2011), which means half of the particles above and half of the particles below this value. For FESEM analysis the granules were fixed with 2% glutaraldehyde overnight at 4 °C after washing with phosphate buffer (pH 7.0) and then was dehydrated with ethyl alcohol and dried (Wang et al., 2007). Sludge volume index (SVI_{30}) (mL $gTSS^{-1}$) was determined by collecting one litre of mixed liquor during aeration phase from the reactor in a graduated cylinder (American Public Health Association-APHA, 2005). After 30 min of settling time, the volume of settled sludge was measured. The ratio of settled sludge volume (mL) to the mass of total suspended solids (g) determined the SVI.

Granule settling velocity (GSV) (m h^{-1}) was determined by free settling test as described by Yu et al. (2009). Randomly ten granules were collected from the reactor and taken in one litre graduated cylinder filled with tap water. Average time taken by individual granule to settle in the cylinder was noted and settling velocity was calculated from distance travelled divided by average settling time of ten granules.

The extracellular polymeric substances (EPS) of the granules were extracted by a heat extraction procedure (Li and Yang, 2007). 50 mL sludge was taken during aeration phase from the SBR and centrifuged for 5 min at 4000 rpm and the pellet was washed twice with supernatant and suspended in 0.2% NaCl solution up to 50 mL. The sludge suspension was kept in the water bath for 30 min at 60 °C. Then the sludge was again centrifuged for 15 min at 4000 rpm. The collected supernatant was considered as EPS extract. EPS was the sum of polysaccharides (PS) and proteins (PN). Polysaccharides were estimated by