Effect of Cycling Time Strategy on The Treatment of Industrial Latex Wastewater with Granular Sludge

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Abstract. Effects of cycling time on the performance of granular sludge in sequencing batch reactors (SBR) were evaluated. Two reactors, reactor 1 (R1) and reactor 2 (R2) was introduced to industrial wastewater with varying chemical oxygen demand (COD) loadings from as low as 0.01 to 0.28 kgCOD/m3/d. However granules in R1 utilizes cycling time of 24 hours whilst R2 utilizes cycling time of 4 hours. The overall performance for both reactors were excellent, where COD removal efficiency was kept at about 78 % to 98 % removal. Although higher COD removal was observed for both reactors, granulation did not occur in R1 when applying cycling time of 24 hours. In contrast to R2, decrement of cycling time to 4 hours promotes the growth of dense and structured sludge granules.

1. Introduction
Treating industrial latex wastewater can be difficult due to varying contaminant level of latex wastewater [1]. Latex processing pose higher contamination due to chemicals added for concentrating process [2]. Rubber effluent contain high level of ammonia and various nutrients creating conventional biological treatment seems irrelevant. Moreover, the production of latex products generate large amount of water. Adding more demands in search of practical and viable technology suitable for treating varying pollution level in latex wastewater particularly treatment within industrial compound with limited area.

Cycling time has been closely associated to directly influenced granulation formation by selecting sludge particles that are able to settle at a given time and consequently resulted in washed out of slow settled sludge flocs[3-4]. Although cycling time directly affected the progression of granules development, the importance of cycling time for granulation formation was not well documented. Since most of the studies focussed on treatment of synthetic wastewater with constant COD level to control the granules progression in the reactor [4-5].

Thus, this study attempt to develop granules with different cycling time strategy using industrial latex wastewater. Selection of cycle time was first carried out by applying 4 hours cycle length to R1 and 24 hours to R2. Cycle time of 4 hours were selected since 3 hours is considered to be unsuitable
workable cycle length as supported by Cydzik-Kwiatkowska [6]. Granulation will not occurred at an extensive cycle time as the sludge only exists in the form of sludge flocs [6]. He further stated, utilization of shorter cycle time of 3 hours will destroy the process formation of granules due to leach out of nitrifying bacteria. To establish a suitable cycle length, cycle time of 4 and 24 hours were compared for removal efficiency of organic and nitrogen compounds. As cycle time promotes microbial growth, consequently resulted in formation of dense and structured granules followed by efficient removal of both organic and nitrogen removal.

2. Materials and methods

2.1. Reactor design

Two cylindrical shaped SBR (R1 and R2) was utilized to cultivate sludge granules. Both reactors had similar reactor configuration with a diameter of 0.05 m. To attain aerobic condition in the reactor, all two reactors were aerated through an air diffuser in order to produce fine air bubbles that were installed at the bottom of the reactor. Flow rate of 5 L/min was introduced to all bioreactors and as a result gave superficial air velocity (SAV) value of 4.25 and 1.1 cm/s. R1 and R2 had a working volume of 1 L.

2.1.1. Setting up of bioreactor

Wastewater was fed to the reactors during “Fill” using dosing pumps, whereas diffuser controlled by aeration pump was installed at the bottom part of the bioreactor as shown in Figure 1. To achieve a stable source of aeration, gas flow meter was fixed between aeration pump and diffuser. Fine bubbles were introduced by a porous stone and the sequential operation of the reactor was automatically controlled by timers.

After treatment, effluent was withdrawn from a port positioned at half the reactor height, effluent is discharged 50 cm from above the reactor’s bottom (50% volumetric exchange ratio). Similar setting up of bioreactors were applied, except for organic loading and reactor design. pH value throughout the investigation was not controlled and temperature of reactors were kept at a room temperature of 28±2°C.

![Figure 1. Schematic diagram of SBR](image-url)
2.2. Inoculated seed sludge
R1 and R2 were started up using activated sludge taken from Shorubber (M) Sdn. Bhd. Wastewater Treatment Plant (WWTP). The seeding sludge shows a fluffy, irregular flocs and loose structured morphology. The seed sludge was taken from an activated sludge storage tank of Shorubber WWTP. Reactor utilized approximately 800 mL of activated sludge which had a mixed liquor suspended solids (MLSS) concentration of 6.57 g/L, sludge volume index (SVI) of 50 mL/g and settling velocity of 0.19 m/h. The seeding sludge shows a fluffy, irregular flocs and loose structured morphology.

2.3. Analytical procedures
Industrial latex wastewater was collected from Shorubber (M) Sdn. Bhd. WWTP storage pond twice a week and stored at 4-6°C. Industrial latex wastewater was then directly introduced into the reactor without adjusting pH, although wastewater was left outside to obtain room temperature of 28±2°C. The COD level of wastewater collected was in the range of 30 mg/L to 978 mg/L.

2.4. Analytical procedures
Influent and effluent from both reactors were analyzed for COD, NH₄⁺, NO₂⁻, NO₃⁻ and PO₄³⁻ using test kits (DR 2800, Hach Lange and Shimadzu ion chromatography). Dissolved oxygen (DO) was measured using DO meter whilst pH is measured using bench-top pH meter. Sludge sample for mixed liquor suspended solids (MLSS), mixed liquor volatile suspended solids (MLVSS) were analyzed according to APHA standard method 2005.

To determine granule size, wet-sieving method was applied by washing the granules several times with phosphate buffer before sorting it according to sizes [11]. Stainless steel sieves having mesh openings of 0.20, 0.60, 0.85, 2.00 and 4.74 mm was utilized for size distribution analysis. Sample was collected from the bottom port of the reactor and was deposited on the 4.74 mm mesh opening sieve and washed with tap water followed by the subsequent sieve size.

Sludge volume index (SVI) of 5, 10, 30 were conducted by observing mixed sludge sample settles at a settling period of 5, 10 and 30 min respectively. Settling velocity (SV) was measured by recording the time taken for an individual granule to fall from a certain height using a measuring cylinder [11]. Changes in morphology and development of the granule size distribution are observed by an Image Analysis (IA) system. IA system consist of computer Olympus Zoom microscope and digital camera.

3. Results and Discussions

3.1. Performance of reactors during granulation
In order to establish a suitable cycle length, this section utilized cycle time of 4 and 24 hours for comparison of COD removal efficiency. Since cycle time promotes microbial growth, that will resulted in formation of dense and structured granules followed by efficient removal of COD. Figure 2, showed the reactor performance during the operational periods of preliminary experiment for cycle time selection between 4 and 24 hours.
Figure 2. COD removal during granulation

Figure 2 shows, COD removal efficiency was kept at 78 to 98% for R1 (4 hours) and 87 to 99% for R2 (24 hours). Although, the average COD removal efficiency for R2 is slightly higher than R1, volume of clean water generated in R1 was 6 times higher than in R2. COD removal in R1 are considered to be satisfactory with only 4% difference than in R2. Highest COD removal efficiency was observed on day 4 at a maximum influent COD level. This can be explained by the ability of microorganisms to easily degrade a simple form of organic substrates[6]. However, towards the end of the experiment COD removal begin to reduce slightly. This is due to the growth of large granules as suggested by Li [4]. As granules size increases the degree of substrate diffusion reduces causes reduction of COD removal [3, 7].

In order to investigate the removal of nitrogen compounds (organics), conversion of ammonium should be monitored (Figure 3). Since nitrification process indicate the presence of nitrifying bacteria in the reactor [7-9]. Complete degradation of ammonium resulted in formation of nitrite, followed with further oxidation of nitrite to nitrate [7].

Figure 3. Percentage of NH$_4^+$-N removal by aerobic granules

Figure 3 showed ammonium removal efficiency was fairly stable in R2 (24 hours) during the first few days. This indicates that all the ammonium exist in the influent was degraded by nitrifying bacteria consequently converted it to nitrite and nitrate [10-12]. Whilst in R1 (4 hours) ammonium removal was not stable when it reached day 4 till day 10. This is mainly due to loss of nitrifying bacteria in R1 (4 hours) as explained by Cydzik-Kwiatkowska [6]. Nitrifying bacteria also known as slow growing nitrifiers favours a longer cycle time since it has to compete with heterotrophs [6]. Shorter cycle time
together with high COD concentration resulted in a prominent heterotroph's growth that are able to surpasses the growth of nitrifying bacteria in the reactor. It has also been demonstrated in Figure 3, an average ammonium removal for 4 hours cycle time is 86 % whereas cycle time of 24 hours achieved average ammonium removal of 93 %. Ammonia uptake in cycle time of 4 hours are lower than cycle time of 24 hours, consequently resulted in lower ammonium removal in R1 (4 hours cycle) [7]. Result depicted the growth of nitrifiers were abundant during cycle 1 compared to cycle 2 due to application of longer cycling time.

Increment of COD value during the operational time can also be one of the reason for reduction ammonium removal in R1 (4 hours). As COD level increases, nitrification was delay during the react since nitrifiers do not compete well for oxygen with heterotrophs [13]. The growth rate of nitrifying was much slower than that of heterotrophic bacteria so the reaction rate of NH\textsubscript{4}+-N to NO\textsubscript{2}--N and NO\textsubscript{3}--N was slow resulting in reduction of removal efficiency [7, 14]. These findings are possible explanation for reduction of ammonium in cycle time of 4 hours on day 5, 9, 16 and 17.

3.2 Effect of cycling time on granulation formation

This section investigate the effect of utilization of inconsistent influent (COD level) on the development of granules by examining its size distribution throughout experimental investigation. Figure 4 showed volume fraction of granules from day 0 to 35 with the application of cycle time of 4 hours since cycle time of 24 hours failed to achieve granulation, with sludge exist in the form of flocs.

R1 (4 hours cycle time) achieved granulation with granule size of $> 4.75$ mm on day 35, granules cultivated in R1 consist of dense structured granules that assist a rapid separation of effluent and granular sludge within shorter time compared to 24 hours cycling time.

4. Conclusions

Industrial latex wastewater was applied to this study for the development of granules. Sudden fluctuation of influent characteristics (COD level) does not affect the granules development. However, although it does not seem to have a great impact compared to cycling time, this study demonstrated that inconsistent COD level from the influent did slightly affected the formation of granules, but only at a certain amount of period that is before sludge flocs reaches complete granulation. Therefore, this shows that COD level influence the granulation time instead of granulation development.

Reaction rate by granules depend on the physical structure of granules. The bigger and denser granules resulted in slower reaction rate due to mass transfer limitation in granule [9], it has been proven by the slight decrease in COD removal rate. The result suggested that large size granules may not be considered as the main goal for granulation if effluent quality is considered. In conclusion,
application of aerobic granules in SBR using industrial latex wastewater (inconsistent COD level) is possible, however selection of cycle time proves to be more significant than the influent itself.

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References
[1] Wang W and Gong J 2011 Front Chem Sci Eng. 5 p 2-10
[2] Najihah A R, Farrah Aini D, Fahmi M R, Ong S A, Latif A and Rani A 2012 Development of aerobic granular in rubber industry wastewater
[3] Iyagba M A, Adoki A and Sokari T G 2008 Afr. J. Agric. Res. 3 p 448-454
[4] Chen F Y, Liu Y Q, Tay J H, Ha and Ning P 2011 J. Hazard. Mater. 189 p 342-8
[5] Li A J, Li X Y and Yu H Q 2011 Process Biochem. 46 p 2269-76
[6] Villain M and Marrot B 2013 Bioresour. Technol. 128 p 134-144
[7] Cydzik-Kwiatkowska A, Zielińska M, Bernat K, Wojnowska-Baryła I and Truchan T 2013 Chemosphere 90 p 2208-15
[8] Yuan X and Gao D 2010 J. Hazard. Mater. 178 p 1041-1045
[9] Guo J, Peng Y, Yang X, Gao C and Wang S 2013 Chemosphere 91 p 68-75
[10] Qin L and Liu Y 2006 Chemosphere 63 p 926-33
[11] Bao R, Yu S, Shi W, Zhang X and Wang Y 2009 J. Hazard. Mater. 168 p 1334-40
[12] Gao D, Liu L and Wu W M J. Hazard. Mater. 186 p 320-7
[13] Liu Y Q, Kong Y, Tay J H and Zhu J 2011 Sep. Purif. 82 p 190-6
[14] Cassidy D P and Belia E 2005 Water Res. 39 p 4817-4823
[15] Chen X, Ni B J and Sin G 2019 Biotechnol. bioeng. 116 p 1280-91