Bioresource Technology 161 (2014) 155–161

Contents lists available at ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

# Characteristics and performance of aerobic granular sludge treating rubber wastewater at different hydraulic retention time

Noor Hasyimah Rosman, Aznah Nor Anuar *, Shreeshivadasan Chelliapan, Mohd Fadhil Md Din, Zaini Ujang

*Institute of Environmental and Water Resource Management (IPASA), WATER Research Alliance, Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia*

## HIGHLIGHTS

- Characteristics and performance of granules were assessed under different HRTs.
- Different HRT led to differences in morphology of microorganisms in the granules.
- Low HRT (i.e. 6 h) enhances the organic and nitrogen removal in granular SBR.
- High $Ca^{2+}$ and $Mg^{2+}$ concentration in aerobic granules was observed at low HRT (6 h).

## ARTICLE INFO

*Article history:*
Received 4 December 2013
Received in revised form 9 March 2014
Accepted 12 March 2014
Available online 21 March 2014

*Keywords:*
Aerobic granular sludge
Hydraulic retention time
Rubber wastewater
Sequencing batch reactor

## ABSTRACT

The influence of hydraulic retention time (HRT, 24, 12, and 6 h) on the physical characteristics of granules and performance of a sequencing batch reactor (SBR) treating rubber wastewater was investigated. Results showed larger granular sludge formation at HRT of 6 h with a mean size of 2.0 ± 0.1 mm, sludge volume index of 20.1 mL $g^{-1}$, settling velocity of 61 m $h^{-1}$, density of 78.2 g $L^{-1}$ and integrity coefficient of 9.54. Scanning electron microscope analyses revealed different morphology of microorganisms and structural features of granules when operated at various HRT. The results also demonstrated that up to 98.4% COD reduction was achieved when the reactor was operated at low HRT (6 h). Around 92.7% and 89.5% removal efficiency was noted for ammonia and total nitrogen in the granular SBR system during the treatment of rubber wastewater.



## 1. Introduction

Natural rubber, an elastic hydrocarbon polymer, originally derived from a milky colloidal suspension or latex of *Hevea brasiliensis*. Malaysia is one of the world's largest natural rubber producers, whereby the rubber industry is an economically significant one. The processing of raw natural rubber can be divided into two types of processes; the production of latex concentrate and the production of Standard Malaysian Rubber (SMR) (Sulaiman et al., 2010). SMR is the current bulk of Malaysian rubber produced in the form of technically specified crumb rubber. Large quantities of effluent were produced from the processing of raw natural rubber since it required huge amount of water for its operation. In general, rubber wastewater has high concentration of ammonia, BOD, COD, nitrate, phosphorus and total solids. If high level of nitrogen and ammonia is discharged to water bodies, it could contributes to undesirable eutrophication and lead to death of some aquatic organisms living in the water. A number of methods especially biological treatment processes, such as anaerobic-cum-facultative lagoon system, anaerobic-cum-aerated lagoon system, aerated lagoon system and oxidation ditch system have been developed for the treatment of rubber wastewater (Sulaiman et al., 2010; Xin et al., 2013). These systems are inexpensive, but required large space, longer effluent treatment period, odor problems, and high operating and maintenance costs. As a result, these circumstances lead to frequent non-compliance to the legal discharge limits (Mohammadi et al., 2010).

Research into aerobic granular sludge technology applications in the real-industrial wastewater has been initiated by previous researchers. This technology offers a small footprint compared to conventional activated sludge based systems since aerobic granular sludge have the advantages of regular and compact structure, good settling properties, high biomass retention and strong ability to withstand high-strength wastewater and shock loadings (Adav

* Corresponding author. Tel.: +60 607 5531125/31738; fax: +60 607 5531575.
*E-mail addresses:* nhasyimah9@gmail.com (N.H. Rosman), aznah@utm.my (A. Nor Anuar).

http://dx.doi.org/10.1016/j.biortech.2014.03.047
0960-8524/

et al., 2008a; Beun et al., 1999; de Kreuk et al., 2007; Gao et al., 2011; Khan et al., 2013; Liu et al., 2009; Liu and Tay, 2004; Tay et al., 2001). Aerobic granular sludge has been extensively reported using sequencing batch reactor (SBR) system fed with various organic substrates including industrial wastewaters. To date, aerobic granular sludge has been successfully tested using wastewater, especially from rubber industry (Rosman et al., 2013).

Aerobic granulation is a complex process influenced or controlled by several factors and mainly dependent on reactor configuration, environmental and operational conditions. The operational parameters including substrate loading rate, aeration intensity, feast–famine regime, settling time and hydraulic retention time (HRT) may be manipulated within the laboratory scale granulation SBR to actively select for stable aerobic granular sludge formation. Studies have shown that a proper HRT should be judiciously selected and carefully maintained for optimization of the reactor performance (Fang and Yu, 2000, 2001). HRT exerts a profound influence on the hydraulic conditions and contact time among different reactants within the reactor. Beun et al. (1999) proposed that a short HRT is favorable for aerobic microbial granulation. Nevertheless, a very short cycle time would suppress the growth of suspended solids due to frequent washout of the suspended material. Pan et al. (2004) cultivated aerobic granules at four different HRTs (2, 6, 12, and 24 h) and reported the differences in the characteristics of aerobic granules formed. They claimed that an HRT of 6 h is most appropriate for biogranulation since the granules possessed higher cell hydrophobicity which indicates the positive roles in promoting biomass settleability and aggregation.

As mentioned previously, aerobic granular sludge appear to be capable of treating the rubber wastewater, however, their performance under different range of HRT is still unknown. Thus, this study sought to investigate the relationship between the granules properties and reactor performance at different HRT applied to the reactor system.

## 2. Methods

### 2.1. Reactor set-up

The reactor had an internal diameter of 8 cm and an effective height of 36 cm. The column of the reactor was made up using borosilicate glass, operated with 1.8 L working volume. A programmable logic controller (PLC) was installed with Zelio-Soft version software to control the actuation of the pumps; influent, effluent, and aerator in the sequencing batch reactor (SBR). The influent wastewater entered through bottom of the reactor column. Fine air bubbles were supplied by means of air bubble diffuser placed at the bottom of the reactor column for aerobic stage. The effluent was withdrawn through the outlet ports positioned at mid-height of the reactor column yielding a volumetric exchange rate (VER) of 50%.

### 2.2. Wastewater characteristics

The rubber wastewater was supplied by a rubber factory located at Kulai, Johor, Malaysia. It was collected weekly and stored in cold storage room at a temperature of 4 °C, in order to prevent the wastewater from undergoing biodegradation due to microbial action. The rubber wastewater contains a small amount of uncoagulated latex, serum with substantial quantities of proteins, carbohydrates, sugars, lipids, carotenoids, as well as inorganic and organic salts and also includes washings water. These substances are readily biodegradable and it will result in high oxygen consumption upon discharge of wastewater in receiving surface water which contributed to high level of COD, BOD and SS. The characteristics of the rubber wastewater was as follows: COD, 1820 mg $L^{-1}$; $BOD_5$, 830 mg $L^{-1}$; total nitrogen (TN), 250 mg $L^{-1}$; ammoniacal nitrogen (AN), 59 mg $L^{-1}$; suspended solids (SS), 270 mg $L^{-1}$, and pH, 7.4.

### 2.3. Seed sludge preparation

The SBR was seeded with domestic sludge taken from an aeration tank of a local, extended aeration-type sewage treatment plant. The sludge was sieved to pass 1.0 mm mesh to remove debris and small particles. The sludge was acclimatized for 6 weeks using domestic and rubber wastewater. During the first 2 weeks, about 900 mL activated sludge was anaerobically cultured with 900 mL of a mixed wastewater containing 2:1 (v/v) of domestic and rubber wastewater. 900 mL of supernatant was removed every 3 days, and then 900 mL of new mixed wastewater was replaced. For the next 2 weeks the ratio of mixed wastewater used was 1:1 and in the last 2 weeks the ratio of mixed wastewater used was 0:1.

### 2.4. Analytical methods

Sample analysis included mixed liquor suspended solid (MLSS), mixed liquor volatile suspended solid (MLVSS), COD, $BOD_5$, $NH_3$, and total nitrogen (TN), all according to Standard Methods (APHA, 2005). COD was analyzed using DR5000 Spectrophotometer (HACH). MLSS content was determined by oven drying of sample at 105 °C for 1 h, whereas MLVSS was determined by ashing the dry sample at 550 °C in a muffle furnace for 15 min. The pH and DO were continuously monitored with the electro probe sensors inserted in the reactor and recorded by a pH/DO meter (Orion 4-Star Benchtop pH/DO Meter).

The granules developed in the SBR column were analyzed for their physical characteristics including sludge volume index (SVI), settling velocity (SV), biomass density and granular strength. Granular sludge settling ability was evaluated in terms of SVI and SV. The SVI procedure was carried out according to the procedure described by de Kreuk et al. (2005). The settling velocity was measured by recording the average time taken for an individual granule to fall from a certain height in a measuring cylinder filled with tap water. The biomass density was determined according to the method described by Beun et al. (1999). Determination of the granular strength was based on Ghangrekar et al. (2005). The granular strength was expressed as the integrity coefficient (%), which is defined as the ratio of residual granules to the total weight of granules sludge after 5 min of shaking at 200 rpm on an orbital platform shaker. The integrity coefficient (IC) indirectly represents the strength of the granules. The lower the IC value, the higher the strength of granules is. The morphological and structural observations of granular sludge were conducted periodically by using a stereo microscope equipped with digital image analyzer (PAX-ITv6, ARC PAX-CAM). The microstructure and microbial compositions within the granules were observed with scanning electron microscope (FESEM-Zeiss Supra 35 VPFESEM). For the pre-treatment procedure for SEM image, the granules were left dried at room temperature prior to gold sputter coating (Biorad Polaron Division SEM Coating System). The mineral content of the granules such as $Ca^{2+}$, $Mg^{2+}$, $Fe^{2+}$, $Al^{3+}$, and $Na^{+}$ was determined using Perkin Elmer Analyst 400 Flame Atomic Absorption Spectrophotometer (FLAA).

### 2.5. Experimental procedures

The mode of operation of the reactor was based on sequencing batch reactor (SBR) system. The SBR cycle time consists of fill