



## Short Communication

## Cultivation of aerobic granular sludge for rubber wastewater treatment

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## HIGHLIGHTS

- ▶ Aerobic granular sludge showed excellent settling ability and sludge volume index.
- ▶ A COD removal rate of 96.5% was observed at an OLR of 3.7 kg COD m<sup>-3</sup> d<sup>-1</sup>.
- ▶ Ammonia and total nitrogen removal efficiencies were 94.7% and 89.4%, respectively.

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## ABSTRACT

Aerobic granular sludge (AGS) was successfully cultivated at  $27 \pm 1$  °C and pH  $7.0 \pm 1$  during the treatment of rubber wastewater using a sequential batch reactor system mode with complete cycle time of 3 h. Results showed aerobic granular sludge had an excellent settling ability and exhibited exceptional performance in the organics and nutrients removal from rubber wastewater. Regular, dense and fast settling granule (average diameter, 1.5 mm; settling velocity, 33 m h<sup>-1</sup>; and sludge volume index, 22.3 mL g<sup>-1</sup>) were developed in a single reactor. In addition, 96.5% COD removal efficiency was observed in the system at the end of the granulation period, while its ammonia and total nitrogen removal efficiencies were up to 94.7% and 89.4%, respectively. The study demonstrated the capabilities of AGS development in a single, high and slender column type-bioreactor for the treatment of rubber wastewater.

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## 1. Introduction

Rubber is one of the main agro-based industrial sectors that play an important role in Malaysia's economy. Presently, Malaysia is the fourth largest rubber producer in the world after Thailand, Indonesia and India (Vijayaraghavan et al., 2008). There are two types of processes in raw natural rubber processing; the production of latex concentrate and the Standard Malaysian Rubber (SMR) (Sulaiman et al., 2010). SMR is the current bulk of Malaysian rubber which is 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. The effluent typically 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 from

the various processing stages (Mohammadi et al., 2010). Both biological and chemical methods are used to treat the rubber wastewater.

Research into aerobic granular sludge technology applications in the real-industrial wastewater has been initiated by previous researchers. The applicability of aerobic granulation in treating a wide variety of industrial wastewater under laboratory scale conditions like abattoir (Yilmaz et al., 2008), dairy (Wichern et al., 2008), livestock (Kishida et al., 2009), winery (López-Palau et al., 2009), chemical industrial wastewater (Liu et al., 2011), palm oil mill effluent (Abdullah et al., 2011), landfill leachate (Wei et al., 2012) as well as industrial effluents (Val del Río et al., 2012) were also been investigated. To date, there is no reported study on the use of aerobic granular sludge system for the treatment of wastewater from rubber industry.

Hence, aerobic granular sludge technology seems to be a reasonable option for studying rubber wastewater treatment. In this study, the process of aerobic granulation in a Standard Malaysian Rubber (SMR) wastewater treatment system were examined by focusing on the feasibility of aerobic granular sludge in treating

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rubber wastewater, as well as the investigation of organics and nitrogen removal in the granular system. This work could be useful for further understanding of the aerobic granulation mechanism as well as for the development of granule-based systems in treating industrial wastewaters.

## 2. Methods

### 2.1. Experimental procedures and bioreactor set-up

Experiments were carried out in a cylindrical column bioreactor (internal diameter of 5 cm and total height of 35 cm) with a working volume of 600 mL. During the start-up period, 300 mL of sludge from a municipal sewage treatment plant was added into the bioreactor system as inoculums, resulting in an initial mixed liquor suspended solid (MLSS) of 5300 mg L<sup>-1</sup> in the bioreactor. A programmable logic controller (PLC) was operated to control the actuations of the pumps; influent pump, effluent pump, and aerator pump with the setting time for each phase in the sequential batch reactor (SBR) mode. The bioreactor was operated in SBR mode at a cycle of 3 h: 5 min of feeding without stirring, 155 min of aerobic reaction, 15 min of settling, 3 min of effluent withdrawal, and 2 min of idling. During the filling phase, wastewater was introduced through ports located at the bottom of the bioreactor. While fine air bubbles for aeration were supplied by means of air bubble diffusers placed at the bottom at a volumetric flow rate of 0.12 m<sup>3</sup> h<sup>-1</sup> (1.70 cm s<sup>-1</sup> superficial air flow velocity). The effluent was withdrawn through the outlet ports positioned at medium height in the column bioreactor, resulting in volumetric exchange ratio (VER) of 50%. The sludge retention time was set by the discharge of suspended solids with the effluent. The bioreactor was operated at room temperature (27 ± 1 °C) and without oxygen and pH control.

### 2.2. SMR wastewater characteristics and seed sludge

The SMR wastewater was obtained from the Chip Hong Rubber Sdn. Bhd., Johor, Malaysia. It was collected from the rubber factory plant once a week and stored in cold storage room at a temperature of 4 °C. Prior feeding into the reactor, the pH was adjusted to a level within neutral condition in the range of 7.0 ± 0.5. The characteristics of SMR wastewater used throughout the experiment were presented in Table 1, in comparison with SMR wastewater as described in the literature (Vijayaraghavan et al., 2008). The seed sludge was taken from an aeration tank of a local, municipal wastewater treatment plant. It had a MLSS concentration of 9.6 g L<sup>-1</sup> and a sludge volume index (SVI) of 84.7 mL g<sup>-1</sup>.

### 2.3. Analytical methods

Measurements of the parameters such as mixed liquor suspended solid (MLSS), mixed liquor volatile suspended solid

(MLVSS), COD, BOD<sub>5</sub>, NH<sub>3</sub>, total nitrogen (TN) and sludge volume index (SVI) were carried out according to Standard Methods for the Examination of Water and Wastewater (APHA, 2005). The pH and DO were continuously monitored with the sensors inserted in the bioreactor and being recorded by a pH/DO meter (Orion 4-Star Benchtop pH/DO Meter). The SVI procedure was carried out according to the procedure described by de Kreuk et al. (2005). 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 compositions within the granule 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).

## 3. Results and discussion

### 3.1. Aerobic granular sludge formation and morphology observation

Under microscopic examination, the initial seed sludge showed a fluffy, irregular, loose-structure morphology and had relative abundance of filamentous microorganisms. The sludge color gradually changed from dark brown to yellowish brown at the end of the experimental period. In the initial stage of granulation (first week), the loose flocs were easily broke up into small pieces if placed under vigorous shaking.

Over the next seven weeks, the flocs-like sludge gradually disappeared and was replaced by small granules with 0.7 mm in average diameter. The flocs became denser under high shear force which induces the biomass aggregates to secrete more exopolysaccharides (Dulekgurgen et al., 2008). The evolution of seed sludge from flocs to granules was achieved due to the interactions between inter-particle bridging process among EPS, microbial cells and ion (Sheng et al., 2010). EPS has the ability to increase the cell hydrophobicity and alter the surface charges on the microorganisms (Zhu et al., 2012), which in turn promote microbial cell adhesion and granulation.

Subsequently, the small granules changed to more regular in shape and gradually increased in size in the following weeks, while more flocculent sludge washed out from the bioreactor, resulting in the accumulation of the aerobic granules with high settling velocity. Finally, mature granules formed after ten weeks of inoculation, leading to a stable operation of the bioreactor. The compact mature granules were smooth with a solid surface, and the average diameter is 1.5 mm. Towards the end of the experiment, granules kept growing in a much lower speed up.

The outer morphology and inner structures of the aerobic granules was further examined using SEM. The SEM observation showed that the granules had a round shape with a clear boundary outline and compact structure. A closer examination revealed that the granules consisted of a wide variety of non-filamentous cocc-shaped bacteria which were tightly linked one another to form the compact structure. Meanwhile, the presence of multiple cavities could also be observed between the clumped bacteria. These cavities were believed to enhance the transport of substrate and oxygen into the inner cores of the granules and metabolic products transfer in and out of the granules (Gao et al., 2011).

### 3.2. Biomass profile and settling properties of granules

Fig. 1 shows the MLSS, MLVSS and SVI variation in the SBR system from the start-up until the end of the study. At the beginning of the experiment, most of the sludge was washed-out from the bioreactor causing a rapid decreased in the biomass concentration

**Table 1**  
Characteristics of raw Standard Malaysian Rubber process wastewater.

Parameters <sup>a</sup>	SMR values (this study)	SMR concentration <sup>b</sup>
pH	7.35	7.5
COD	1850	2960
BOD <sub>5</sub>	890	1380
Suspended solids (SS)	270	310
Total nitrogen (TN)	248	n.s. <sup>c</sup>
Ammoniacal nitrogen (AN)	49	57

<sup>a</sup> All other parameters are in mg L<sup>-1</sup> except pH.

<sup>b</sup> Source: Vijayaraghavan et al. (2008).

<sup>c</sup> n.s. = Not specified.

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