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2012

Performance evaluation of the pilot-scale static granular bed reactor (SGBR) for industrial wastewater treatment and biofilter treating septic tank effluent using recycled rubber particles

Jin Hwan Oh *Iowa State University*

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## **Performance evaluation of the pilot-scale static granular bed reactor (SGBR) for industrial wastewater treatment and biofilter treating septic tank effluent using recycled rubber particles**

by

## **Jin Hwan Oh**

A dissertation submitted to the graduate faculty

in partial fulfillment of the requirements for the degree of

# DOCTOR OF PHILOSOPHY

Major: Civil Engineering (Environmental Engineering)

Program of Study Committee: Timothy G. Ellis, Major Professor Hans van Leeuwen Shih Wu Sung Thomas Loynachan Raj Raman

Iowa State University

Ames, Iowa

2012

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#### **EXECUTIVE SUMMARY**

The performance and operational stability of the three pilot-scale SGBR for the treatment of industrial wastewater were investigated in this study. High organic removal efficiencies (over 94% of COD removal) were obtained from the two pilot-scale SGBR (R1 and R2) for the treatment of slaughterhouse wastewater. During the operation of reactors, the solid retention times over 240 and 150 days for the R1 and R2, respectively were obtained. The pilot-scale SGBR was also successfully employed for treating dairy processing wastewater under psychrophilic conditions. COD, BOD, and TSS removal rates obtained were 93, 96, and 90%, respectively, even at low temperatures of 11°C. The SGBR achieved average COD, BOD, and TSS removal efficiencies higher than 91% even at high loading rates up to 7.31 kg  $\text{COD/m}^3/\text{d}$ with an HRT of 9 h. The of three pilot-scale SGBR were operating in a stable condition since pH values were in the optimal range and VFA/alkalinity ratios were fairly low throughout the experimental period. The average methane yield of  $0.26$  L CH<sub>4</sub>/g COD<sub>removed</sub> was possibly affected by a high fraction of particulate COD and operation at low temperatures. In addition to the conversions of soluble COD into methane, particulate organic matter was physically retained by adsorption to granular sludge and the entrapment of coarse suspended solids in the sludge bed. Increased headloss through the granular bed due to the accumulated excess biomass and the retained solids were controlled by periodic backwashing.

A proper backwash rate is necessary to ensure effective removal of dispersed fine sludge and excessive suspended solids. Assuming that the average granule size and density in this study are in the range of 0.8-1.6 mm and 1000-1060 kg/m<sup>3</sup>, respectively, the minimum backwash rates varied from 0.02 to 4.34 m/h depending on the size and density of the granules. The proper



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backwash velocity ranged from 0.11 to 11.33 m/h based on the assumption that the bed porosity increased up to 0.4 and 50% expansion was selected as the optimum value. Therefore, backwash at a flow rate of 10-15 gpm (3.91-5.87 m/h) was applied to the pilot-scale SGBR (cross-sectional area: 6.25 ft<sup>2</sup>) treating dairy wastewater in Tulare, CA.

Performance of the lab-scale RRP biofilter was compared to a conventional gravel system and a peat biofilter system for treatment of septic tank effluent. During the study, the RRP biofilter provided similar or better performance than other systems in terms of organic removal and hydraulic capacity. After the start-up period, RRP biofilter achieved removal efficiencies for BOD5, TSS, ammonia nitrogen of 96, 93, and 90%, respectively, over the range of hydraulic loading rates of 1.4 to 5.0 gpd/ft<sup>2</sup>. On the other hand, the peat biofilter failed hydraulically and the gravel system showed high TSS concentrations in the effluent. RRP provided high surface area and sufficient time for biological treatment. In addition, RRP provided a non-toxic media for biofilm attachment in biofilter. RRP was observed to provide ammonia adsorption capacity. The results showed that RRP has the potential to be used as substitutes for natural aggregate such as gravel in septic system drainfields. The RRP biofilter can be used as alternative septic systems for the sites where an existing septic system has failed or site conditions, such as high groundwater table or small lot size, are not suitable for the installation of conventional septic systems.



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### **CHAPTER 1. GENERAL INTRODUCTION**

#### **Anaerobic treatment**

The anaerobic degradation of complex organic matter is carried out by multistep chemical and biological process. Complex and particulate organic matters such as proteins, carbohydrates, and lipids are decomposed into simpler soluble compounds (amino acids, glucose, and long chain fatty acids) by hydrolysis. Hydrolysis is carried out by extracellular enzymes excreted by hydrolytic and fermentative bacteria. Hydrolysis is usually considered to be a ratelimiting step of the overall anaerobic digestion process. During Acidogenesis, the hydrolyzed compounds are fermented into volatile fatty acids (VFA), also referred to as short-chain fatty acids (SCFA), such as acetate, propionate, and butyrate. Short-chain fatty acids except acetate are degraded to acetate,  $H_2$ , and  $CO_2$  by hydrogen producing acetogenic bacteria. About 66% of long chain fatty acids is oxidized to acetate and  $33\%$  to  $H_2$ . Acetate is also directly derived from acidogenic fermentation of amino acids and sugars, and homoacetogenesis, in which  $H_2$  is used to reduce  $CO<sub>2</sub>$  to acetate by hydrogen consuming acetogenic bacteria. In the final step of anaerobic digestion process, acetate is converted into  $CO<sub>2</sub>$  and  $CH<sub>4</sub>$  by acetoclastic methanogenesis. Approximately 70% of the total methane formed in anaerobic digestion originates from acetate and the other 30% is produced from reduction of  $CO<sub>2</sub>$  by hydrogenotrophic methanogens (hydrogen oxidizing methanogens). Proton-reducing acetogenic bacteria is not suppressed by excessive  $H_2$  level due to syntrophic association between hydrogenproducing acetogenic bacteria and hydrogen-utilizing methanogenic bacteria to maintain a low H<sup>2</sup> partial pressure. On the other hand, both methane-producing bacteria and sulfate-reducing bacteria compete for the same electron donor, acetate and  $H_2$ . Sulfate-reducing bacteria may



outcompete methanogens under low acetate conditions because methanogens have a lower affinity for acetate than sulfate-reducing bacteria.



## **Figure 1−1. Anaerobic degradation of complex organic matters**

- 1) Fermentative bacteria
- 2) Hydrogen-producing acetogenic bacteria
- 3) Hydrogen-consuming acetogenic bacteria
- 4) Hydrogenotrophic methanogens  $(CO<sub>2</sub>-reducing$  methanogens)
- 5) Acetoclastic methanogens



#### **Granular Sludge**

Immobilization of biomass without a support material was first observed in upflow anaerobic sludge bed (UASB) reactors through the formation of sludge granules (Lettinga *et al*., 1980). MacLeod *et al*. (1990) proposed a layered structure model for anaerobic granules developed in UASB reactors based on the microscopic observations. The outer layer contains mainly heterogeneous populations together with acidogens and hydrogen-consuming microorganisms. Hydrogen-producing acetogens and hydrogen-consuming microorganisms predominated in the middle layer and the core dominated by acetotrophic methanogens (*Methanosaeta* spp.). Several studies reported that the bacterial composition and the structure of granular sludge were affected by the type of substrate (Fang *et al*., 1994; Grotenhuis *et al*., 1991).



**Figure 1−2. Layered structure of anaerobic granules (MacLeod** *et al***., 1990)**

Henze (2008) and Schmidt and Ahring (1996) reported that common characteristics of methanogenic granular sludge as listed in Table 1−1.





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**Table 1−1. Characteristics of granular sludge**

<sup>a</sup>Henze *et al*. (2008)

<sup>b</sup>Schmidt and Ahring (1996)

#### **Static Granular Bed Reactor (SGBR)**

The static granular bed reactor (SGBR) is a simple downflow high rate anaerobic system developed at Iowa State University (U.S. Patent No. 6,709,591). The main advantages for the SGBR are high organic removal efficiency and operational simplicity. Due to the downflow configuration of the SGBR, the system has a simpler inlet flow distribution design and the generated biogas is easily separated from the granules and wastewater effluent and collected at the top of the reactor as shown in Figure 1−1. As the influent wastewater is mixed with the bulk liquid by the countercurrent flow of biogas and liquid, high concentrations of organics in the influent wastewater are dispersed and diluted. The downflow operation also allows solids in the influent to be filtered through the granular bed. Biogas-induced mixing sufficiently reduces dead volumes and short-circuiting and eliminates the need for mechanical agitation and mixing systems or recirculation pumping. The SGBR utilizes a bed of active anaerobic granules for treatment of wastewater with relatively small reactor volume sizes. Therefore, the SGBR can reduce relatively high costs associated with the packing materials, mixing equipment, or recirculation systems required. The high concentration of biomass retained within the reactor



allows the contact between the dissolved organic matter and the active biomass to be maximized, and an extremely long solids retention time (SRT) can be achieved. In addition, the suspended solids are trapped in the granular bed for a sufficient period to allow hydrolysis followed by further degradation to occur.

The SGBR has been shown to be capable of treating a variety of wastewaters at high organic loading rates and short HRT in numerous laboratory and pilot scale studies (Debik *et al*., 2005; Evans and Ellis, 2005; Evans and Ellis, 2006; Evans and Ellis, 2007; Mach and Ellis, 2000; Park *et al*., 2012; Roth and Ellis, 2004). The performance of the SGBR fed with a synthetic wastewater composed of sucrose and non-fat dry milk was compared to the UASB reactor. At an HRT of 8 h, the COD removal efficiencies of the SGBR and UASB reactor were 91 and 78%, respectively (Evans and Ellis, 2010). Roth and Ellis (2004) reported that the SGBR treating pork slaughterhouse wastewater obtained average COD removal efficiency greater than 90% at an OLR range between 1.9 and 4.55 kg COD/m<sup>3</sup>/d. Park *et al.* (2012) also investigated the performance of a pilot-scale SGBR treating slaughterhouse wastewater. The reactor showed stable treatment efficiency at fluctuating organic loading rates from 0.77 kg/m<sup>3</sup>/d to 12.76 kg/m<sup>3</sup>/d and achieved COD removal efficiencies above 95%. Rapid start-up (less than one month) was observed in both SGBR reactors. They concluded that increased OLR coupled with reduced HRT only slightly affected performance of the SGBR. Debilk and Coskun (2009) reported that the SGBR treating poultry slaughterhouses wastewaters attained average COD removal rates of 95%. Debik *et al*. (2005) also investigated the SGBR performance in treating leachate and obtained more than 90% COD removal rates efficiency at a high organic loading rate of 15 kg/m<sup>3</sup>/d.





**Figure 1-3. Schematic diagrams of SGBR**

#### **Industrial wastewater**

Slaughterhouses and meat processing wastewater typically contains blood, fat, and manure, resulting in high content of organic matter (US-EPA, 2002). The suspended and colloidal matter in the form of fats, proteins, and cellulose may have detrimental effect on the performance of anaerobic reactors due to their insolubility and slow rate of degradation (Johns, 1995; Torkian, 2003). Aerobic treatment processes are considered less suitable for slaughterhouse wastewater due to high energy consumption for aeration, large quantities of



sludge production, and oxygen transfer limitations (Gavala *et al*., 1996; Rajeshwari *et al*., 2000; Speece, 1996). Therefore, anaerobic biological processes have been employed to treat slaughterhouse wastewater with high organic loads. Anaerobic lagoons are widely used for the treatment of primary treated slaughterhouse wastewater due to low operational and maintenance cost. On the other hand, the disadvantages of lagoons include odor problem and the large land area requirement. Therefore, high rate anaerobic processes have been proposed as alternatives to anaerobic lagoons, including the anaerobic contact (AC), upflow anaerobic sludge blanket (UASB), anaerobic filter processes (AF), and anaerobic sequence batch reactor (ASBR) (US-EPA, 2002; Johns, 1995).

Dairy wastewaters are typically characterized by their high biological oxygen demand (BOD) and chemical oxygen demand (COD) concentrations, resulting from proteins, fats, and carbohydrates including lactose and high levels of nitrogen and phosphorus (Brown and Pico., 1979; Omil *et al*., 2003; Perle *et al*., 1995). Thus, dairy wastewater is regarded as a complex type of substrate. Due to the presence of high organic matter, anaerobic treatment processes are considered suitable for dairy wastewater. Carbohydrates in dairy wastewater are mainly lactose and easily degradable while proteins and lipids are less biodegradable. However, lipids may cause inhibitory effects on anaerobic processes as it is hydrolyzed to glycerol and long chain fatty acids (LCFAs). Long chain fatty acids were reported to cause inhibition in methane production.



#### **Study Objective**

The aim of this research was to evaluate performance and operational stability of the three pilot-scale SGBR systems treating dairy processing wastewater and slaughterhouse wastewater, and to determine the optimum backwash parameters in order to achieve proper solids control. The kinetics of the two pilot-scale SGBR systems treating slaughterhouse wastewater were determined and kinetic models were compared to apply for describing the substrate utilization of the SGBR. In order to determine kinetic coefficients, mathematical models including Monod kinetics, Grau second-order model, and Stover-Kincannon model were applied to the system. Finally, in an unrelated investigation, the performance of biofilter system using a recycled rubber particle (RRP) system was also compared to a conventional gravel system and a peat system to demonstrate the feasibility of RRP as biofilm support media.

#### **Dissertation Organization**

This dissertation is organized into four major parts with individual papers. The first part evaluates performance and operational stability of the SGBR treating dairy processing wastewater. The second part proposes optimum backwash procedures. The third part is the determination of kinetic parameters for the SGBR treating slaughterhouse wastewater. The final part demonstrates the feasibility of a recycled rubber particles (RRP) as biofilm support media in bioreactors for treating septic tank effluent.



# **CHAPTER 2. DAIRY PROCESSING WASTEWATER TREATMENT BY ON-SITE PILOT STATIC GRANULAR BED REACTOR (SGBR)**

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# **Introduction**

The dairy industry is considered to be one of the largest sources of industrial wastewater. This situation will continue as the demand for dairy products increases. Dairy processing effluents are mainly generated from cleaning of transport lines and equipment between production cycles, cleaning of tank trucks, washing of milk silos, and equipment malfunctions or operational errors (Danalewich *et al*., 1998; Eroglu *et al*., 1991; Perle *et al*., 1995). Dairy processing wastewaters are typically characterized by their high biological oxygen demand (BOD) and chemical oxygen demand (COD) concentrations resulting from proteins, fats, and carbohydrates, including lactose, and high levels of nitrogen and phosphorus. Also included are various cleaning and sanitizing agents. Combined, these compounds result in the potential for environmental problems in terms of high organic load on the local municipal sewage treatment systems (Brown and Pico., 1979; Omil *et al*., 2003; Perle *et al*., 1995).

Anaerobic treatment processes are regarded as suitable methods for treating dairy wastewater due to their advantages for treating industrial wastewaters with higher biodegradable organic matter and the characteristics of the dairy wastewater. Aerobic treatment processes, on the other hand, require high energy consumption for aeration and generate large amounts of sludge (Gavala *et al*., 1996; Rajeshwari *et al*., 2000; Speece, 1996). Therefore, laboratory-scale



anaerobic reactors for dairy wastewater treatment have been investigated in a number of previous studies. A typical range organic loading rate (OLR) for high rate anaerobic digesters including upflow anaerobic sludge blanket (UASB) reactors, anaerobic filters (AF), anaerobic sequencing batch reactor (ASBR) was 2.0 to 15 kg COD/m<sup>3</sup>/d (Demirel *et al.*, 2005). The laboratory-scale UASB reactors for treatment of combined dairy and domestic wastewater achieved COD and TSS removal rates of 69 and 72% at an hydraulic retention time (HRT) of 24 h and an OLR range between 1.9 and 4.4 kg  $\text{COD/m}^3/\text{d}$  (Tawfik *et al.*, 2008).

The static granular bed reactor (SGBR) is a simple downflow high rate anaerobic system developed at Iowa State University (U.S. Patent No. 6,709,591). The advantages for the SGBR include operational simplicity and high quality effluent. Due to the downflow configuration of the SGBR, it has a simpler inlet flow distribution design and the generated biogas is easily separated from the granules and wastewater effluent and collected at the top of the reactor. There are no packing materials and no mixing equipment or recirculation systems required, resulting in lower capital and operating costs. The SGBR utilizes a bed of active anaerobic granules for treatment of wastewater with relatively small reactor volume sizes, which contribute to higher COD removal efficiencies and biomass concentration of the granules. The SGBR has been shown to be effective in laboratory and pilot studies on treatment of municipal wastewater, and landfill leachate (Debik *et al*., 2005; Mach and Ellis, 2000; Roth and Ellis, 2004). In previous research, the performance of the SGBR treating a synthetic wastewater composed of sucrose and non-fat dry milk was compared to the UASB reactor. At an HRT of 8 h, the COD removal efficiencies of the SGBR and UASB reactor were 91 and 78%, respectively (Evans and Ellis, 2005).



The Industrial Wastewater Treatment Plant (IWTP) at the city of Tulare, California treats wastewater from dairy processing industries that produce cheese, butter, ice-cream, and other dairy-based products. Industrial wastewater is treated by an anaerobic bulk volume fermenter (BVF) followed by a series of partially aerated facultative ponds. The existing IWTP with a capacity of 7.1 million gallons per day (MGD) is being expanded to comply with present and future discharge regulations and to handle additional flows and loadings from the various manufacturers. Therefore, a more robust and cost-effective wastewater pretreatment system is required to treat the unique and high-strength wastewater. The aim of this study was to observe the performance of a pilot-scale SGBR treating wastewaters from dairy processing plants. The performance of the SGBR was monitored and analyzed in terms of COD removal efficiencies and variation of volatile fatty acids (VFA). The pilot-scale SGBR was demonstrated under various operational conditions to develop the full-scale design parameters.

#### **Materials and methods**

#### *Wastewater source and characteristics*

Dairy processing wastewater is composed of easily degradable carbohydrates, mainly lactose, as well as proteins and lipids which are less biodegradable. Approximately, 4.4 million gallons per day (mgd) of industrial wastewater was being generated from various industrial sources including seven large dairy processing plants. Thus, dairy processing wastewater used in this study can be considered as a complex type of wastewater. Although the composition of the wastewater with respect to carbohydrates, proteins and lipids was not determined in this study,



the wastewater might be expected to contain a high percentage of lipids according to the average particulate COD/VSS ratio of  $2.77 \pm 0.86$  g COD/g VSS. The average ratio of pCOD to VSS was estimated based on total COD, soluble COD, and VSS concentrations, and the ratio was similar to the stoichiometric conversion factors for lipid of 2.87 g COD/g VSS. The characteristics of dairy processing wastewater used in this study are given in Table 2–1. The ratio of BOD<sub>5</sub> to COD was calculated to evaluate the potential biodegradability of the organic contents in dairy processing wastewater. Dairy wastewater with a ratio below 0.40 can be considered recalcitrant due to the presence of non-milk constituents (Danalewich *et al*., 1998).

Parameter	Value
pH	$5.79 \pm 0.67$
TSS, mg/L	$493 \pm 196$
VSS, mg/L	$486 \pm 196$
Total COD, mg/L	$2883 \pm 631$
Soluble COD, mg/L	$1629 \pm 286$
$BOD_5$ , mg/L	$1637 \pm 423$
Biodegradability (BOD <sub>5</sub> /COD)	$0.6 \pm 0.2$

**Table 2−1. Characteristics of dairy processing wastewater**

#### *Reactor set-up and operation*

A pilot-scale SGBR made of stainless steel was installed at the industrial wastewater treatment plant (IWTP) in Tulare, California and operated for 6 months. The reactor had a total volume of 2,200 gallon and a working volume of 1,500 gallon (Figure 2**−**1). The reactor was seeded with 900 gallons of anaerobic granules (60% of the reactor working volume) obtained from an operating UASB at City Brewing Company in La Crosse, Wisconsin. Specific methanogenic activity of the seed granular sludge was  $0.333$  g COD-CH<sub>4</sub>/g VSS/d. The anaerobic granules were transferred using a progressive cavity pump to prevent the disintegration of the granules. The dairy wastewater stream was pumped into a 2,500 gallon feed tank that was



used to store an influent wastewater for feeding the SGBR, to separate settleable and floating solids from the wastewater, and to adjust the pH of the wastewater by addition of sodium hydroxide (NaOH). The wastewater was sampled from the influent channel, and it was assumed that there was no significant change in the COD or TSS through the feed tank. This was verified by testing the SGBR influent against the influent channel. These two sampling points had similar average COD and TSS concentrations ( $p = 0.155$  and 0.647). The dairy wastewater from the tank was fed into the SGBR using a progressive cavity pump and distributed through semi-circular pipe installed in the upper part of the reactor. A feed inlet pipe was also used for the drainage of backwashed water from the granular bed. The underdrain system consisted of perforated PVC pipes used for effluent discharge and backwashing, and a gravel layer was used to prevent biomass washout and protect underdrain pipes from clogging. The treated effluent was discharged by gravity through the outlet pipe equipped with 8 valves having different height positions from 5 ft to 12 ft to control the water level in the reactor. The biogas was collected through a PVC pipe installed at the top of the digester. The biogas was subsequently fed into the gas scrubber filled with a mixture of coarse and fine steel wool to remove hydrogen sulfide (H<sub>2</sub>S). The gas treated by the scrubber was measured with a wet-test gas meters (RITTER<sup>®</sup>) drum-type gas meter, Hawthorne, NY). The biogas was also sampled periodically by using Tedlar<sup>TM</sup> bags through the valve installed on the pipe for gas composition analyses. A manometer and a side mounted tubular level indicator were installed to monitor the pressure and water level changes in the reactor. The SGBR system was operated in continuous mode at an HRT of 48 h to maintain the optimum organic loading rate during the start-up period.





**Figure 2−1. Pilot-scale SGBR system in Tulare, CA**





**Figure 2−2. Schematic diagrams of the pilot-scale SGBR system**

#### *Data collection and analytical methods*

Influent and effluent samples were collected and analyzed 4-5 times per week to monitor the performance of the reactor over a period of 6 months. The parameters including pH, total alkalinity, biochemical oxygen demand (BOD), total suspended solids (TSS), and volatile suspended solids (VSS) were determined in accordance with Standard Methods for the Examination of Water and Wastewater (APHA,1998). Samples for SCOD and VFAs were filtered using glass-fiber filters prior to testing (Whatman GF/C, 1.2 µm). Soluble COD and VFA were measured from filtrate. Chemical oxygen demand (COD), soluble chemical oxygen demand



(SCOD), and volatile fatty acids (VFAs) were measured with a colorimeter following the Hach method 8000 and 8196. Biogas production was measured with a  $\text{RITTER}^{\circledast}$  (Hawthorne, NY) wet-test (drum-type) gas meter and the biogas composition was analyzed with a Gow Mac Instrument Company (Bethlehem, PA) Series 350 Thermal Conductivity Detector. The biogas samples were also sent to BSK analytical laboratory in Fresno, CA for gas composition. Specific methanogenic activity tests (SMA tests) were performed to observe changes in sludge activities according to method described by Rinzema *et al.* (1988).

## **Results and discussion**

#### *Performance of the SGBR*

The performance of the SGBR with respect to COD, BOD, and TSS removal efficiencies was evaluated under a wide range of organic and hydraulic loading rates and temperature conditions. Organic loading rates varied in the range of 0.63 to 9.72 kg  $\text{COD/m}^3/\text{d}$  and HRT ranged between 9 to 96 h. The reactor was also operated at ambient temperature (19  $\pm$  5 °C), which is under sub-mesophilic and psychrophilic conditions.

The SGBR was initially operated in continuous mode at an HRT of 48 h to allow the granules to acclimate to the substrate. However, headloss increased in the reactor after 16 days of operation as a result of the accumulation of large particles since the raw wastewater prior to pretreatment was fed to the reactor. Therefore, a feed tank was installed with a screening process to trap debris and remove floating matter from the influent on day 23. Despite the increase in headloss, the SGBR showed good performance in terms of COD and TSS removal during the first 23 days as shown in Figure 2**−**3. The average COD and TSS removal efficiency were 92 and



80%, respectively. Longer HRTs (96 and 72 h) and an average OLR less than 0.9 kg  $\text{COD/m}^3/\text{d}$ were temporarily maintained from day 23 to day 37. During this period, improvements in TSS reduction and operational stability in terms of head loss build up were observed. As the performance of the SGBR remained stable during the start-up period, the OLR was gradually increased by a stepwise decrease in HRT.

Stable effluent COD concentrations were observed in the SGBR, even with the fluctuating influent COD levels ranging from 2000 to 7340 mg/L throughout the study as shown in Figure 2**−**3. The average total and soluble effluent COD concentrations were 160 and 89 mg/L, respectively, corresponding to both total and soluble COD removal rates more than 94%. The SGBR achieved average BOD removal of 97%, which might be due to relative biodegradable nature of the wastewater having BOD to COD ratio of 0.6.

After the feed tank installation, suspended solids reduction improved and 96% TSS removal was obtained at an HRT of 36 h. However, elevated levels of suspended solids were observed at an HRT of 30 h and thus the fluctuation of effluent TSS removal efficiency tended to decrease. Although effluent COD also slightly fluctuated, removal efficiencies were maintained between 87 to 96%. Considering the influent TSS concentration, it did not seem to be the main cause of the increase in effluent TSS. The decreased TSS removal possibly resulted from the incomplete hydrolysis of particulate organic matter. The results indicated that there was a trend in the ratio of pCOD to tCOD in the effluent which increased with increasing organic loading rate resulting from the shortening the HRT. A decrease in temperature may have contributed to this effect. Increased hydraulic shear forces could have reduced the retention time of influent



TSS in the SGBR. Consequently, bacteria would utilize the readily biodegradable soluble COD. Hydrolysis of particulate COD which is facilitated through extracellular enzymes may have been limited at the shorter HRT and lower temperature conditions. The SGBR did not have heating and insulation and was exposed to a sudden change in temperature from day 75 (21°C) to day 86 (11°C). This could have affected the stability and performance of the SGBR system because the various metabolic groups of microorganisms involved in the digestion process might respond differently to reduced temperature. The hydrolysis of the particulate matter is very sensitive to temperature and usually considered to be the rate-limiting step. Hence, the reduced hydrolysis rate could cause the decrease in the degradable fraction of organic matter and consequently lead to an accumulation of particulate organic matter in the SGBR during operation at low temperatures (below 15°C) for 36 days (Lettinga *et al*., 1983). The increase in head loss was also observed during this period as entrapped solids were accumulated. Sanz and Fdz-Polanco (1990) reported accumulation of suspended solids at the top of the anaerobic fluidized bed reactor (AFBR) treating municipal sewage under lower temperature conditions ( $10^{\circ}$ C). Uemura and Harada (2000) also reported entrapment or accumulation of suspended solids in the upflow anaerobic sludge bed (UASB) reactor for the treatment of raw domestic sewage at 13°C. Several studies have suggested that longer HRT was required to provide sufficient time for microorganism to solubilize biodegradable particulate at low temperatures (Elmitwalli *et al*., 2002; Zeeman and Lettinga, 1999). Accordingly, the SGBR was operated at longer HRT (48, 42, and 36 h) for 17 days (day 86-103) to allow microorganisms to acclimate to the lower temperature (11°C). During this period, COD removal rate was maintained at around 93% and TSS removal efficiencies fluctuated around 90%. Even at high loading rates up to 7.31 kg  $\text{COD/m}^3$ /d with an HRT of 9 h, high COD removal and TSS efficiencies more than 94 and 89%



were accomplished, respectively. Lower temperature and high loading rates did not appear to have a detrimental effect on the SGBR performance in terms of COD and suspended solids removal efficiencies. Suspended solids in the effluent did not significantly depend on the variations observed in the influent probably due to the removal through the physical process of suspended solids retention in the sludge bed. This indicates that the SGBR has a high capacity of retaining solids and acts in a filtration capacity due to its downflow operation.



**Figure 2−3. Variation of COD and TSS concentrations with removal efficiency**



#### *Monitoring parameters and the stability of the SGBR*

The pH, alkalinity, volatile fatty acids, and ammonia were monitored to evaluate the operational stability of the SGBR and control the system if necessary. The use of various acid or alkaline cleaning and sanitizing agents and other chemicals in the dairy industry resulted in influent pH values ranged from 4.7 to 8.6 with an average of 5.8. In the feed tank, fluctuating influent pH values were stabilized and adjusted by the addition of a 49% sodium hydroxide solution. As shown in Figure 2−4, the effluent pH was stably maintained between 6.7 and 7.9 with an average 7.24, which was within the optimal pH range between 6.5 and 8.2 for methane production (Speece, 1996). It was shown that the alkalinity decreased from 875 to 575 mg/L and VFA concentrations increased from 18 to 54 mg/L as HRT decreased from 48 to 30 h during the coldest period (day 94 to 104). The increase in solubility of  $CO<sub>2</sub>$  could result in consuming alkalinity under psychrophilic conditions. As the hydraulic and organic loading rate further increased, the increase in VFA production might have resulted in a rapid consumption of alkalinity in the system. Hence, alkalinity and VFA concentrations were maintained at around 533mg/L and 40 mg/L, respectively. These observations are supported by stable pH values in the effluent. In other words, alkalinity was used for maintaining stable pH conditions for methanogens, and hydrogen and volatile organic acids degrading methanogens in the SGBR were not inhibited due to enough buffer capacity, thereby resulting in no VFA accumulation. The ratio of VFA to alkalinity, indicating process stability, was monitored to ensure proper digestion condition (Ripley *et al*., 1986). A VFA to alkalinity ratio less than 0.3 reflects stable operating conditions, while a ratio between 0.3 and 0.4 indicates a potential for upset and possible need for corrective action. If the ratio exceeds 0.8, the process may fail as a result of digester acidification and inhibition of methanogens by VFA accumulation (WPCF Manual of Practice No. 16, 1987).



Figure 2−4 depicts the results of the ratio of VFA to alkalinity in the effluent. The ratio ranged from 0.02 to 0.12 through the study. The ratio of intermediate alkalinity to partial alkalinity (IA/PA) was also suggested by Ripley *et al*. (1986) as a simple and useful indicator of digester stability because VFA measurement was not required. PA is the titration from the pH of the original sample to an end-point of pH 5.75 and IA is related to VFA presence and the titration from a pH of 5.75 to 4.3. A ratio of IA/PA below 0.3 is recommended for anaerobic digestion, and the ratio of IA to PA in this study was on average 0.24. Therefore, it can be concluded that the SGBR system was operating in a stable condition since the pH was in the optimal range and VFA/alkalinity ratios were fairly low throughout the experimental period.

The concentration of total ammonia in the effluent was measured to monitor the possibility for ammonia toxicity. It has been reported that ammonia concentrations below 200 mg/L could be beneficial to anaerobic microorganisms (Liu and Sung, 2002). However, high free ammonia concentration may inhibit the methanogenic activity, which is a function of temperature and pH (Hobson and Shaw, 1976; Liu and Sung, 2002; McCarty, 1964; Vandenburg h and Ellis, 2002). Total ammonia concentrations in the effluent were relatively low and ranged from 8 to 104 mg/L as N, with an average of  $56 \pm 22$  mg/L as N. The maximum concentration of free ammonia was found to be less than 2 mg/L since the SGBR was operating under neutral pH conditions and low temperatures.





**Figure 2−4. Variation of pH, alkalinity, VFA/ALK ratio, and IA/PA ratio**

0 20 40 60 80 100 120 140 160 180

Operation time, Days

Ĉ

#### *Conductivity*

0.00 0.05 0.10 0.15

IA/PA

The electrical conductivity (EC) of the raw wastewater and the SGBR effluent over time was monitored. With caustic soda addition for pH adjustment, the average EC increased by 200



0 0.02 0.04 0.06 to 1200 μS/m. The caustic soda used for alkalinity resulted in an overall increase in EC through the system, but the resultant increase is only a fraction of that for caustic soda.

#### *Specific methanogenic activity*

Specific methanogenic activity (SMA) tests were conducted to determine the maximum methane production rate of anaerobic granular sludge under controlled environmental conditions. The methanogenic activity of biomass is expressed as the COD equivalent of the methane produced per gram of VSS per day (g COD-CH4/g VSS-d). The methanogenic activity of granular sludge can vary depending on operational parameters including HRT, OLR, process temperature, mixing conditions, influent COD concentration, substrate characteristics, adaptation of the biomass, presence of inhibiting factors, and reactor configuration (Grotenhuis *et al*., 1991; Kato *et al*., 1997; Kettunen and Rintala, 1997; Lettinga, 1995).

Specific methanogenic activity of the seed granular sludge was  $0.333$  g COD-CH<sub>4</sub>/g VSS-d and the granular sludge was sampled from the two sampling ports, located in the middle and bottom of the reactor (1.2 and 0.6 m from the base), to compare the activity of sludge at different depths. The SMA of the granules sampled from the middle and bottom of the reactor was slightly lowered to 0.270 and 0.288 g COD-CH<sub>4</sub>/g VSS-d, respectively, on day 86 at an OLR of 1.70 kg COD/ $m^3$ /d. During the first 85 days of operation, average values of OLR and influent COD concentration were 1.53 kg  $\text{COD/m}^3/\text{d}$  and 2799 mg/L, respectively. Therefore, the effect of substrate concentration on the activity could be considered negligible. The decrease in the methanogenic activity was probably due to the effect of changed operational conditions such as operating temperatures on the SMA because OLR and influent COD concentrations were fairly



constant. Ho and Sung (2010) reported that acetoclastic methanogenic activity of suspended sludge in laboratory-scale anaerobic membrane bioreactors (AnMBRs) at 15 °C was shown to be 40% lower than at 25 °C after 75 days of operation. The value of the half-saturation constants (KS) of acetate has been found to increase at decreasing temperatures (Lin *et al*., 1987). Therefore, the lower methanogenic activity than the seed sludge was most likely related to the decreased activity of acetoclastic methanogens due to the lower substrate affinity for acetate after exposure to low temperature (11°C) conditions. On the other hand, the population of hydrogenotrophic methanogens (hydrogen oxidizing methanogens) might increase due to the increase in  $H_2$  and  $CO_2$  level in the reactor at low temperatures, which was expected to contribute to methane production. The proliferation of hydrogenotrophic methanogens at low temperature has been reported in several previous studies (Collins *et al*., 2005; Conrad and Wetter, 1990; Enright *et al*., 2005; Kotsyurbenko *et al*., 1996; Lettinga *et al*., 1999; Lettinga *et al*., 2001; McHugh *et al*., 2004). However, there was an insignificant decrease in acetoclastic methanogens activity, indicating that methanogens showed ability to adapt to low temperature conditions.

The SMA of the granules from the middle and bottom increased to 0.478 and 0.337 g COD-CH4/g VSS-d, respectively, on day 125 at an HRT of 24 h. The increase of the SMA might have resulted from elevated temperature (18°C). This would indicate that the activity of acetoclastic methanogens was recovering from temperature shock. The highest acetoclastic methanogenic activity was observed in the middle part of the SGBR. It should be noted that additional backwash through the side valve from day 93 might provide sufficient mixing to enhance the contact between methanogens and substrate, and lead to selective wash out of finely



dispersed sludge in the middle part of the SGBR. The differences in SMA values could be explained by concentration gradients of substrate within the granular bed, different concentrations of methanogenic populations, or different substrate affinity of methanogens. For example, *Methanosaeta* has a higher substrate affinity (thus lower K<sub>s</sub>) for acetate but longer doubling times than *Methanosarcina*. Accordingly, *Methanosaeta* will be the dominant acetoclastic methanogens at low acetate concentrations, while the fast growing *Methanosarcina* is usually favoured by high acetate concentrations due to its shorter doubling times. Kalyuzhnyi *et al*. (1996) reported the population of methanogens in the lower part of the laboratory-scale UASB reactor was 2-3 orders of magnitude higher than in the upper part since VFA levels decreased with increasing reactor height. Ruiz *et al*. (1997) also found that lower methanogenic activity in the upper part of the UASB operated at 37°C due to the accumulation of inert solids.

Several studies have reported that the population of acetoclastic methanogens, as well as its activity, decreased with increasing OLR and decreasing HRT (Fang and Yu, 2000; Jawed and Tare, 1996; Kalyuzhnyi *et al*. 1996). It is possible that an accumulation of slowly biodegradable substrate in the sludge bed could lead to deterioration of the SMA under high loading conditions, or shorter HRT may somewhat limit methanogens by washing out the available substrate (Elefsiniotis and Oldham, 1994; Sayed *et al*, 1987). On the other hand, the SMA values observed in this study were above 0.3 g COD-CH<sub>4</sub> g<sup>-1</sup> VSS<sup>-1</sup> day<sup>-1</sup> at higher OLR and an HRT of 12 h.

The results obtained in the SMA tests were found to be in the range reported in previous studies, even though anaerobic systems were treating various wastewaters under different



operating conditions (Table 2−2). From the results of the SMA tests, the methanogens in the SGBR have shown the capacity to withstand organic and hydraulic shock loads.

Reactor	Original feed	Operating Temperature $({}^{\circ}C)$	Test temperature $({}^{\circ}C)$	<b>SMA</b> $(gCH4-COD/gVSS-d)$	Reference
<b>EGSB</b>	Synthetic wastewater	20	20	0.5	Yoochatchaval et al (2008)
<b>UASB</b>	Synthetic wastewater	35-37	35	$0.117 - 0.709$	Kalyuzhnyi et al (1996)
<b>UAF</b>	Synthetic wastewater	35	35	0.359	Mohammad and Vinod (1999)
EGSB-AF	Synthetic wastewater	15	37	0.028-0.825	Enright et al (2005)
<b>UASB</b>	Pharmaceutical wastewater	$30 - 36$	35	0.182	Ince <i>et al.</i> $(2001)$
<b>TPAD</b>	Mixture of primary and waste activated sludge	35	35	$0.092 - 0.418$	Vandenburgh and Ellis (2002)
EGSB-AF	<b>Brewery</b>	15	37	0.95	Connaughton et al. (2006)
AnMBR	NFDM, acetate, starch	25	25	0.172	Ho and Sung (2010)
AnMBR	NFDM, acetate, starch	15	25	0.103	Ho and Sung (2010)
<b>SGBR</b>	Slaughterhouse wastewater	$24 - 26$	35	$0.324 - 0.377$	Park et al. (2012)
<b>SGBR</b>	Dairy processing wastewater	$11 - 20$	35	$0.270 - 0.478$	This study

**Table 2−2. Comparison of acetoclastic SMA results in different processes**

UASB: Upflow Anaerobic Sludge Blanket, UAF: Upflow Anaerobic Filter, EGBR: Expanded Granular Bed Reactor AF: Anaerobic Filter, TPAD: Temperature Phased Anaerobic Digestion, AnMBR: Anaerobic Membrane Bioreactors



#### *Biogas production and composition*

The biogas collected from the top of the digester fed into a gas scrubber to remove hydrogen sulfide (H2S), and then treated biogas was measured with a wet-test gas meter. The biogas was also sampled periodically by using Tedlar<sup>TM</sup> bags through the valve installed on the pipe for gas composition analyses. The measured biogas volume was converted to the volume at standard temperature and pressure (STP) condition (0°C, 1 atm). The dissolved methane in the effluent and backwash water were determined in accordance with Henry's law and included in actual methane production. Typically, the percentage of methane in the biogas increases while that of carbon dioxide  $(CO<sub>2</sub>)$  decreases as operating temperature is lowered because methane is much less soluble than  $CO<sub>2</sub>$ . However, in this study there was no obvious increase in the proportion of methane in the biogas with decreasing temperature. From the results of the biogas composition and production, an average methane content of 75% was obtained and the amount of methane dissolved in the effluent and released during backwashing was 7.1 and 14.9%, respectively, of the total methane production.




**Figure 2−5**. **Effect of temperature and OLR on methane production**

The actual methane production rate  $(L/d)$  and yield  $(L CH<sub>4</sub>/g COD<sub>removed</sub>$  at STP conditions) were compared with the theoretical value as shown in Figure 2−6. The theoretical methane production rate was calculated based on the assumption of 94% COD removal efficiency, 90% COD removed conversion into methane as well as a theoretical methane yield of 0.35 L CH<sub>4</sub>/g COD<sub>removed</sub>. The results showed that methane productions (L/d) were improved by the increase of operating temperatures and OLR (decreasing HRT) from 118 days. Average methane production rate at temperatures below 18°C was 3,119 L/d, and it increased to 3,616  $L/d$  at temperature above 18 $^{\circ}$ C in the same HRT of 24 h. The highest methane production rate was observed at an OLR of 2.8 kg  $\text{COD/m}^3/\text{d}$  and temperature of 19 $\textdegree$ C. However, the difference between actual methane production and the theoretical maximum production increased with increasing OLR. The actual amount of methane accounted for 77% of the theoretical values at an average OLR of 2.0 kg  $\text{COD/m}^3/\text{d}$ , and it decreased to 46% of the theoretical values at an



average OLR of 5.0 kg  $\text{COD/m}^3/\text{d}$ .



**Figure 2−6. The actual and theoretical methane production and yield**

The higher conversion of the wastewater to methane was obtained at lower OLR and relatively high temperatures. The average methane yield from day 34 to 47 was found to be 0.33



L CH<sub>4</sub> /g COD<sub>removed</sub> at an OLR of 1.3 kg COD/m<sup>3</sup>/d and temperature of 23<sup>o</sup>C, which corresponded to 94% of the theoretical value. Conversion to methane of the removed COD decreased with the increase in OLR. Consequently, the overall average methane yield was 0.26 L CH4/g CODremoved. These lower methane yields could possibly be attributed to a high fraction of particulate COD (32 to 52%) and operation at low temperatures. The results also suggested that soluble or particulate organic matter was not completely converted into methane, but were physically retained by adsorption of the colloidal fraction of wastewater to granular sludge and the entrapment of coarse suspended solids in the sludge bed.

Percentage of hydrolysis (H), acidification (A) and methanogenesis (M) were calculated according to the following equations (2.1), (2.2) and (2.3), respectively (Elmitwalli et al., 2002b) and summarized in Table 3. The influent VFA concentration of 147 mg/L as HAc and conversion factor of 1.28 g COD per g VFA were assumed (Danalewich et al, 1998; Rössle and Pretorius, 2001).

$$
H(\%) = 100 \left( \frac{COD_{\text{CH}_4} + sCOD_{\text{eff}} - sCOD_{\text{inf}}}{tCOD_{\text{inf}} - sCOD_{\text{inf}}} \right)
$$
 (2.1)

$$
A(\%) = 100 \left( \frac{COD_{\text{CH}_4} + COD_{\text{VFA eff}} - COD_{\text{VFA inf}}}{tCOD_{\text{inf}}} - COD_{\text{VFA inf}} \right)
$$
\n(2.2)

$$
M(\%) = 100 \left( \frac{COD_{\text{CH}_4}}{tCOD_{\text{inf}}} \right) \tag{2.3}
$$

$$
tCOD = sCOD + pCOD \tag{2.4}
$$

where total  $COD =$  soluble  $COD +$  particulate  $COD$ 

 $tCOD_{\text{inf}}$  = amount of total COD, mg/L

 $sCOD<sub>inf</sub>$  and  $sCOD<sub>eff</sub>$  = amount of soluble COD in influent and effluent, mg/L



 $\textit{COD}_{\text{CH4}}$  = amount produced CH<sub>4</sub> including dissolved form, mg/L

 $\text{COD}_{\text{VFA} \text{ inf}}$  and  $\text{COD}_{\text{VFA} \text{ eff}}$  = amount of VFA in influent and effluent, mg/L

Time	Temperature	<b>OLR</b>	ັ Methane yield	H	A	M
(Days)	$({}^{\circ}C)$	$(kg \text{ COD/m}^3/d)$	$(L CH4/CODremoved)$		(% )	
34-47	23	1.3	0.29	$93 \pm 39$	$36 \pm 7$	$79 \pm 16$
48-61	22	1.8	0.21	$27 \pm 40$	$25 \pm 5$	$59 \pm 11$
$62 - 76$	21	1.6	0.26	$57 \pm 23$	$30 \pm 3$	$71 \pm 6$
77-93	14	1.8	0.22	$-12 \pm 101$	$23 \pm 7$	$52 \pm 15$
94-105	11	1.7	0.21	$14 \pm 44$	$24 \pm 5$	$54 \pm 13$
106-118	13	2.2	0.19	$17 \pm 22$	$20 \pm 3$	$49 \pm 6$
119-135	18	3.4	0.21	$24 \pm 39$	$19 \pm 7$	$51 \pm 18$
136-152	18	3.0	0.26	$54 \pm 64$	$25 \pm 10$	$66 \pm 28$
153-169	20	3.5	0.21	$13 \pm 52$	$20 \pm 6$	$55 \pm 15$
170-185	20	6.6	0.12	$-45 \pm 39$	$10 \pm 2$	$32 \pm 6$
Mean	18	2.8	0.23	$23 \pm 63$	$23 \pm 9$	$56 \pm 19$

**Table 2−3. Hydrolysis (H), acidification (A) and methanogenesis (M)**

The calculated percentages of hydrolysis, acidification and methanogenesis indicated that hydrolysis was more sensitive to low temperature and high loading rate compared to acidification and methanogenesis. The slow hydrolysis of entrapped solids could allow solids to accumulate in the sludge bed at high organic loading rates. Consequently, overall conversion to methane of the removed COD was limited, resulting in lower values of methane yield. Pavlostathis and Giraldo-Gomez (1991) also concluded that the rate of anaerobic conversion of complex organic matter is, in most cases, limited by the hydrolysis step.



#### *COD balance and backwashing*

The principal equation for COD balance of the SGBR is:

$$
tCOD_{\text{inf}} = tCOD_{\text{eff}} + COD_{\text{CH}_4} + COD_{\text{accumulated}} + COD_{\text{backward}}
$$
\n(2.5)

$$
COD_{\text{CH4}} = V_{\text{CH4}} \times \frac{\text{g COD}}{0.35 \text{L CH}_{4}} \tag{2.6}
$$
\n
$$
V_{\text{CH}_4} = V_{\text{measured}} + V_{\text{released}} + V_{\text{dissolved}} = (V_{\text{biogas}} \times \% \, CH_{4}) + V_{\text{released}} + S_{\text{CH4}} (Q_{\text{eff}} + Q_{\text{backwash}}) \tag{2.7}
$$

$$
V_{\text{CH}_4} = V_{\text{measured}} + V_{\text{released}} + V_{\text{dissolved}} = (V_{\text{biogas}} \times \% \ CH_4) + V_{\text{released}} + S_{\text{CH4}} (Q_{\text{eff}} + Q_{\text{backward}})
$$
(2.7)  

$$
S_{\text{CH4}} = 1.4 \times 10^{-3} \frac{\text{mol}}{\text{I} \cdot \text{atm}} \times \exp \left[ 1700 \left( \frac{1}{272.15 \times T} - \frac{1}{209.15} \right) \right] \times 1 \text{atm} \times 22.4 \frac{\text{L}}{\text{mol}}
$$
(2.8)

$$
V_{\text{CH}_4} = V_{\text{measured}} + V_{\text{released}} + V_{\text{dissolved}} = (V_{\text{biogas}} \times \% \ CH_4) + V_{\text{released}} + S_{\text{CH4}} (Q_{\text{eff}} + Q_{\text{backwash}})
$$
(2.7)  

$$
S_{\text{CH4}} = 1.4 \times 10^{-3} \frac{\text{mol}}{\text{L} \cdot \text{atm}} \times \exp \left[ 1700 \left( \frac{1}{273.15 + T} - \frac{1}{298.15} \right) \right] \times 1 \text{atm} \times 22.4 \frac{\text{L}}{\text{mol}}
$$
(2.8)

$$
COD_{\text{backward}} = \left[ \left( VSS_{\text{backward}} - VSS_{\text{biomass}} \right) \times \frac{pCOD_{\text{inf}}}{VSS_{\text{inf}}} \right]
$$
(2.9)

where  $tCOD = SCOD + p COD$ 

 $V_{\text{biogas}}$  = volume of the biogas, L

 $V_{\text{released}}$  = volume of methane released to atmosphere during the backwash

 $\%CH_4$ = methane content of the biogas,  $\%$ 

 $S_{CH4}$  = solubility of methane at STP, L CH<sub>4</sub>/L

 $k_H$  = Henry's Law constant at 298.15*K* = 0.0014 mol L<sup>-1</sup> atm<sup>-1</sup>

 *CODbackwashed* = amount of COD removed by backwash, mg/L

 $VSS_{\text{backward}} =$  amount of VSS in backwash water, mg/L

 $VSS_{\text{biomass}} =$  amount of wasting biomass, mg/L

 $pCOD<sub>inf</sub>/VSS<sub>inf</sub>$  = ratio of particulate COD to VSS in influent, g COD/g VSS

Several assumptions were made to develop the COD mass balance:

(1) 90% of COD removed was converted into methane and the remaining 10% of COD



removed was utilized for biomass synthesis.

- (2) The biomass yield coefficient was  $0.10$  gVSS/gCOD<sub>removed</sub>.
- (3) 1.0 g of COD removed produced 0.35 L of CH4.
- (4) Soluble COD was more readily biodegradable than particulate COD.
- (5) The difference between total  $CH_4$  production and  $CH_4$  from soluble COD conversion represented the increase in soluble COD by hydrolysis of suspended solids.
- (6) The amount of VSS in backwash water included undegraded suspended solids and wasted biomass.



**Figure 2−7. Overall COD balance of the SGBR** 





**Figure 2−8. The accumulation of COD in the SGBR**

The COD mass balance indicated that 50 and 25% of the influent COD were treated by means of the conversion of COD to methane and backwash, respectively, and remaining 19% of the influent COD was retained and accumulated in the reactor. More than 70% of soluble COD and 23% of particulate COD were converted into methane. This indicates that soluble COD was responsible for most of the methane production and some methane was also produced from the hydrolysis and fermentation of entrapped particulate organic matter. There was only a slight accumulation of COD, despite the sudden drop in temperature. On the other hand, the accumulation of COD tended to increase with increasing OLR and decreasing HRT. A gradual accumulation of slow and non-biodegradable solids within the void spaces between the granules caused headloss in the reactor. Therefore, sludge and suspended solids were removed by means of periodic backwashing. Backwashing frequency was determined according to head loss and was usually once a week. The 300 gallons of effluent stored in a 305-gallon tank was pumped at the flow rate of 10 gpm (gallon per minute) for 30 minutes. Approximately, half of accumulated



particulate COD was removed and controlled by wasting of undegraded suspended solids as well as dispersed fine sludge via backwash. In addition to the routine backwash through the underdrain, backwash through side valves at 2 and 4 ft was performed from day 93 due to the dense and compact granular bed. This likely loosened the entire granular bed and removed slow and non-biodegradable solids in the SGBR. Therefore, the accumulation of COD slowed and increased slightly until day 118 even at lower temperatures. The increase in particulate COD accumulation was accompanied by increased OLR while soluble COD was adequately treated. However, a significant accumulation of undegraded organic matter was observed at short HRT of less than 18 h and ORL more than  $3.5 \text{ kg }$  COD/m<sup>3</sup>/d.

## **Conclusions**

The pilot scale SGBR was successfully employed for treating dairy processing wastewater under psychrophilic conditions and high loading rates. At low temperatures of 11<sup>o</sup>C COD, BOD, and TSS removal rates obtained were 93, 96, and 90%, respectively. The SGBR achieved average COD, BOD, and TSS removal efficiencies higher than 91% even at high loading rates up to 7.31 kg  $\text{COD/m}^3/\text{d}$  with an HRT of 9 h. The SGBR system was operating in a stable condition since the pH was in the optimal range and VFA/alkalinity ratios were fairly low throughout the experimental period. The average methane yield  $(0.26 \text{ L CH}_4/\text{g COD}_{\text{removed}})$  could possibly be affected by a high fraction of particulate COD (32 to 52%) and operation at low temperatures. Soluble COD was responsible for most of the methane production and particulate organic matter was physically retained by adsorption of the colloidal fraction of wastewater to granular sludge and the entrapment of coarse suspended solids in the sludge bed. The accumulated excess biomass and the retained solids were removed from the system by means of



periodic backwashing.

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# **CHAPTER 3. BACKWASHING OF THE STATIC GRANULAR BED REACTOR (SGBR)**

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## **Introduction**

In the static granular bed reactor (SGBR), wastewater enters at the top of the reactor through the inlet flow distribution system and passes downward by gravity through the dense bed of active anaerobic granules. The downflow mode of operation allows the influent wastewater to be mixed with the bulk liquid by the countercurrent flow of biogas and liquid. Thus, high concentrations of organics in the influent wastewater are immediately dispersed and diluted. Biogas induced mixing sufficiently reduces dead volumes and short-circuiting and eliminates the need for a mechanical agitation mixing systems or recirculation pumping.

Due to the high biomass concentration, the contact between the dissolved organic matter and the active biomass are maximized. The suspended solids are trapped in the granular bed for a sufficient period to allow hydrolysis followed by further degradation to occur. The SGBR has been shown to be capable of treating a variety of wastewaters at high organic loading rates and short HRT in laboratory scale studies, and it has been successfully employed for pilot scale treatment of meat processing wastewater (Park *et al*., 2012; Roth and Ellis, 2004).



Suspended biomass within the interstitial void spaces was considered to be a significant factor in substrate removal. On the other hand, excessive biomass growth results in a decrease in the available area for the organic matter to diffuse into the granules, and therefore, potentially decreases the removal efficiency. Wastewater containing high levels of suspended solids may cause a gradual accumulation of slow and non-biodegradable solids within the void spaces between the granules. The slow hydrolysis of entrapped solids at low temperatures also results in solids accumulation. Consequently, as the pores become occupied by entrapped solids and biomass, a decrease in the effective porosity will lead to a rapid buildup of head loss, channeling, and short-circuiting of flow through the reactor. Previous studies have reported that the rate of head loss buildup increased with the increase in organic loading (Park *et al*., 2012; Roth and Ellis, 2004). Park *et al*. (2012) reported that the increase in the head loss occurred due to a clogged underdrain system caused by the solids accumulation in the reactor. Therefore, periodic backwashing is required to minimize problems associated with headloss buildup and clogging of the underdrain system. Additionally, the potential mixing effect created by the backwashing process can enhance the contact between the wastewater and the biomass. Although there have been several reports on backwasing method in operation of the SGBR, no information exist on backwashing parameters. In this study, optimum backwash flow rate and bed expansion were determined for proper backwashing and to prevent wash out of sludge granules from the SGBR.

#### **Materials and methods**

#### *Backwashing of the SGBR treating dairy processing wastewater in Tulare, CA*

For the backwash process, the treated effluent from the storage tank (305 gallons) was



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injected through side valves (2 ft from the bottom of the reactor) and underdrain pipes, and evenly distributed over the bottom of the SGBR as shown in Figure 3**−**1. Approximately, 4.7% of the total volume of treated wastewater was used for backwashing. The backwashed water was discharged into the main influent channel of the plant.



**Figure 3−1. Backwashing process**

### *Terminal settling velocity and bed expansion during backwash*

The terminal settling velocity of the granules can be calculated from balancing the gravitational and drag forces exerted on the granules. The particle Reynolds number,  $Re_t$ (dimensionless) and the terminal settling velocity for spherical particles,  $u_t$  (m/h) can be calculated using the following equation:



$$
u_t = \sqrt{\frac{4gd_p(\rho_p - \rho)}{3C_D\rho}}
$$
\n(3.1)

$$
\text{Re}_t = \frac{\rho d_p u_t}{\mu} \tag{3.2}
$$

where *g* is the gravitational acceleration (9.81 m/s<sup>2</sup>),  $d_p$  is the particle diameter (m),  $\rho_p$  and  $\rho$  are the density of particle and liquid, respectively  $(kg/m^3)$ ,  $C_D$  is the drag coefficient (dimensionless), and  $\mu$  is the liquid viscosity (kg/m/s). The granules usually have a spherical form but they are not smooth or rigid, and thus  $C<sub>D</sub>$  for the granules is higher than that of smooth rigid spheres.

Although several correlations have been proposed (Ganguly, 1987; Nicolella et al., 1999; Perry and Green, 1997; Schiller and Naumann, 1935; Yu and Rittmann, 1997), *C<sup>D</sup>* for the granules in the intermediate flow regime  $(1 < Re<sub>t</sub> < 100)$  was estimated by using the following correlation proposed by Ro and Neethling (1990):

$$
C_D = \frac{24}{\text{Re}_t} + 21.55 \,\text{Re}_t^{-0.518} \tag{3.3}
$$

Based on the results of settling velocity, bed expansion during backwashing was estimated by using the empirical equation suggested by Richardson and Zaki (1954):

المشارات

$$
\varepsilon^n = \frac{u}{u_t} \tag{3.4}
$$

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$$
\eta = \frac{H_e - H_0}{H_0} \times 100\%
$$
\n(3.5)

$$
\eta = \frac{\varepsilon - \varepsilon_0}{1 - \varepsilon} \times 100\% \tag{3.6}
$$

where *u* is the backwash velocity (m/h), *n* is the expansion index (dimensionless),  $H_0$  is the initial height of the granular bed (m), and  $H<sub>e</sub>$  is the height of the expanded granular bed (m). The expansion index was determined as a function of the Reynolds number as shown below:

$$
n = 4.65 \t Ret < 0.2
$$
  
\n
$$
n = 4.35Ret-0.03 \t 0.2 < Ret < 1
$$
  
\n
$$
n = 4.45Ret-0.1 \t 1 < Ret < 500
$$
  
\n
$$
n = 2.39 \t 500 < Ret
$$
\n(3.7)

The bed voidage, *ε*<sub>0</sub> for spherical particles usually varies from 0.4 to 0.45, and the bed voidage of 0.4 was used. Substituting Eq. 3.7 into Eq. 3.4, the bed voidage can be written as follows:

$$
\varepsilon = e^{\frac{\ln \frac{u}{u_t}}{4.35 \text{Re}_t^{-0.03}}}
$$
\n
$$
\varepsilon = e^{\frac{\ln \frac{u}{u_t}}{4.45 \text{Re}_t^{-0.1}}}
$$
\n
$$
\varepsilon = e^{\frac{\ln \frac{u}{u_t}}{4.45 \text{Re}_t^{-0.1}}}
$$
\n
$$
1 < \text{Re}_t < 500
$$
\n
$$
(3.9)
$$

#### *The minimum backwash velocity*

The required minimum backwash velocity for fluidization of the granular bed,  $u_{mf}$  in the



SGBR, could be predicted. Galileo number, *Ga* (dimensionless) represented the ratio of viscous and gravitational forces. *Ga* and *umf* were calculated following the equation below (Wen and Yu, 1966):

$$
Ga = \frac{d_p^3 g(\rho_p - \rho)\rho}{\mu^2} \tag{3.10}
$$

$$
u_{mf} = \frac{\mu}{d_p \rho} \Big[ \big( 33.7 \big)^2 + 0.0408Ga \Big]^{0.5} - 33.7 \frac{\mu}{d_p \rho} \tag{3.11}
$$

#### *Head loss in operating SGBR*

The porosity of the granular bed in the SGBR was estimated by using the Kozeny equation and head loss measurements.

$$
\frac{h}{L} = \frac{k\mu}{\rho g} \frac{(1-\varepsilon)^2}{\varepsilon^3} \left(\frac{A}{V}\right)^2 V
$$
\n(3.12)

$$
h = 180 \frac{\mu}{\rho g} \frac{(1 - \varepsilon)^2}{\varepsilon^3} \frac{L}{\psi^2 d^2} V
$$
\n(3.13)

where  $h =$  Head loss, m

- $L =$  Depth of granular bed, m
- $k =$  Dimensionless Kozeny coefficient commonly about 5
- $\mu$  = Viscosity of fluid, kg/m/s
- $\varepsilon$  = Porosity



 $A/v = 6/(v/d) =$  Grain surface area per unit of grain volume, m<sup>-1</sup>

- $V =$  Superficial approach velocity,  $m/s$
- $g =$  Gravitational acceleration, m/s<sup>2</sup>
- $\rho$  = Density of fluid, kg/m<sup>3</sup>
- $\psi$  = Shape coefficient (0.75 assumed)
- *d* = Diameter of granules, mm

## **Results and discussion**

#### *Terminal settling velocity and bed expansion during backwash*

The calculated settling velocities and  $Re<sub>t</sub>$  of the granules with different sizes using the solver function in Microsoft Excel are shown in Figure 3−2.







**Figure 3−2. Estimated settling velocity and Re<sup>t</sup> of the granules**

The average granule size in this study was estimated based on the results of size analysis in previous studies since anaerobic granules were obtained from the same source (operating UASB treating brewery wastewater in La Crosse, Wisconsin) (Mach and Ellis, 2000; Park *et al*., 2012; Roth and Ellis, 2003). Determination of granule size by image analysis was performed in the Materials Analysis and Research Laboratory of the Civil, Construction and Environmental Engineering Department at Iowa State University. Previous studies have reported that the granule size in the range of 0.7−1 mm in early stages of operation increased as the system operated over time. In general, the granules typically have a diameter from 0.5 to 2.5 mm and a density ranging from 1,000 to 1,050 kg/m<sup>3</sup> (Ferry, 1993; Henze *et al.*, 2008). Angelidaki *et al.* (2003) reported that settling velocities of granular sludge were in the range of 18–100 m/h. Figure 3**−**2 shows that the settling velocity varied from 0.1 to 92 m/h depending on the size and density of the granules. Assuming that the average diameter and density of the granules were 1.2



mm and  $1,020 \text{ kg/m}^3$ , respectively, the settling velocity was found to be 19 m/h. Although the granular bed may be expanded to the same extent by lower backwash velocity at lower temperatures since the backwash water is denser, it was also assumed that the temperature of backwash water was 20ºC.

From the results of settling velocity, the bed expansion during backwashing could be predicted using numerical relationships in terms of bed voidage as a function of fluid superficial velocity (Richardson and Zaki, 1954). Although the biogas may lead to more turbulence resulting in detachment of retained solids, several studies have reported that the effect of biogas on the bed expansion can be ignored thus those system were regarded as two phase (solid-liquid) systems (Leitao, 2004; Nicolella, 1999). The bed expansion was plotted against the backwash velocity for different size of granules with identical density of  $1,020 \text{ kg/m}^3$  as shown in Figure 3−3. The predicted bed expansion increased with increasing backwash velocity. At the backwash velocity of less than 0.5 m/h, granules larger than 1.2 mm were not fluidized and remained at static conditions.





**Figure 3−3. Relationship between backwash velocity and bed expansion**

## *The minimum backwash velocity*

The required minimum backwash velocity for fluidization of the granular bed, *umf* in the SGBR could be predicted to ensure adequate cleaning. Figure 3−4 shows that the backwash velocity of 1 m/h was sufficient to fluidize small granules or particles ( $d_p < 0.6$  mm). The minimum backwash rate of 0.67 m/h was required to initiate fluidization of the bed  $(d_p=1.2 \text{ mm})$ ,  $\rho_p$ =1,020 kg/m<sup>3</sup>).





**Figure 3−4. The minimum fluidization velocity for different size and density of the granules**

#### *Head loss in operating SGBR*

The accumulated excess biomass and the retained solids may decrease the volume of void space in the granular bed leading to the rapid development of head loss through the system. Therefore, assuming the SGBR acted as a filter, the porosity of the granular bed in the SGBR was estimated by using the Kozeny equation (Eq. 3.13) and head loss measurements. However, the calculation of head loss using Kozeny equation was only useful to provide an estimation of the minimum head loss since the granular bed was a mixture of different sized granules and the fluid was wastewater. The average granule size of 1.2 mm was assumed for the calculation and a decrease in either granule size or porosity may cause an increase in head loss. The calculated porosity of the granular bed by observed head loss can be used to determine the backwash velocity for achieving optimum bed expansion.





**Figure 3−5. Variations of porosity of the granular bed**

Figure 3**−**5 showed that the porosity of the granular bed varied from 0.11 to 0.47 and the average value was found to be 0.30. The bed porosity increased after backwash, resulting in an average porosity of 0.33 during the period of treatment of 1,500 gallons of wastewater after the backwash was completed.







The required backwash velocity for different porosities to maintain a 50 percent expansion of the granular bed was estimated by using Eq. 3.6 (Figure 3−6). The results showed that as the bed porosity decreased, a moderate backwash rate for a longer duration was required. Also in the same manner, backwash velocity needed to be increased with increasing bed porosity as the accumulated biomass and suspended solids were removed from the bed during backwash.

#### *Backwashing process*

For the backwash process, the influent valve was closed. The treated effluent (305 gallons) from the storage tank was injected through side valves and underdrain pipes, and evenly distributed over the bottom of the SGBR. Lower backwash flow rates are required until the bed is fluidized and the velocity is gradually increased to the desired backwash rate.

## **Conclusions**

A proper backwash rate is necessary to ensure effective removal of dispersed fine sludge and excessive suspended solids. Lower backwash flow rates are required to avoid disrupting the granular bed and the velocity is gradually increased to the desired backwash rate. Assuming that the average granule size and density in this study are in the range of 0.8-1.6 mm and 1000-1060 kg/m<sup>3</sup>, respectively, the minimum backwash rates varied from 0.02 to 4.34 m/h depending on the size and density of the granules. The degree of bed expansion during backwash of granular filtration in water treatment is usually in the range of 20 to 90% of the filter bed length. The proper backwash velocity ranged from 0.11 to 11.33 m/h based on the assumption that the bed porosity increased up to 0.4 and 50% expansion was selected as the optimum value. Therefore,



backwash at a flow rate of 10-15 gpm (3.91-5.87 m/h) was carried out in the pilot study of the

SGBR (cross-sectional area:  $6.25 \text{ ft}^2$ ) treating dairy wastewater in Tulare, CA.

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# **CHAPTER 4. KINETIC MODELING AND PERFORMANCE EVALUATION OF SGBR FOR TREATING MEAT PROCESSING WASTEWATERS**

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# **Introduction**

Slaughterhouses and meat processing plants consume substantial amounts of water ranging from 4.2 to 16.7  $m^3$  per tonne of live carcass weight and 80% of the water is discharged as effluent during the multiple stage of processing. These stages include livestock reception, hide treatment, and cleaning of casings, offal and carcasses (Johns, 1995). Typical wastewater volumes generated from hog slaughterhouses range from 2.0 to 5.1  $m<sup>3</sup>$  per tonne of live weight kill (LWK) with an average of 3.9 m<sup>3</sup> per tonne LWK. Meat processing wastewater typically contains blood, fat, and manure, resulting in high content of organic matter with a mean value of 8.3 kg  $BOD<sub>5</sub>$  per tonne LWK (US-EPA, 2002). The suspended and colloidal matter in the form of fats, proteins, and cellulose may have a detrimental effect on the performance of anaerobic reactors due to their insolubility and slow rate of degradation (Johns, 1995; Torkian, 2003).

A variety of systems have been developed to provide primary, secondary, and tertiary treatment for removal of floating and settleable solids, BOD reduction, and nutrient removal, respectively, from meat processing wastewater. Dissolved air flotation (DAF) is widely used in the primary treatment for removal of suspended solids from the wastewater. Although physical



and chemical processes have been investigated, anaerobic biological processes have remained the preferred method for the treatment of slaughterhouse wastewater with high organic loads. Aerobic treatment processes, on the other hand, are not considered suitable for slaughterhouse wastewater due to high energy consumption for aeration, large quantities of sludge production, and oxygen transfer limitations (Gavala *et al*., 1996; Rajeshwari *et al*., 2000; Speece, 1996). Anaerobic lagoons are extensively used for the treatment of primary treated slaughterhouse wastewater. However, high rate anaerobic processes have been proposed as alternatives to anaerobic lagoons, including the anaerobic contact (AC), upflow anaerobic sludge blanket (UASB), anaerobic filter processes (AF), and anaerobic sequence batch reactor (ASBR) (US-EPA, 2002; Johns, 1995). Sayed *et al*. (1993) evaluated the two stage UASB system for treatment of slaughterhouse wastewater. The two-stage DAF-UASB system achieved 90% COD reduction at an HRT of 10h and an OLR of 4 kg  $\text{COD/m}^3/\text{d}$ , which was proposed as an alternative to the two stage UASB system (Manjunath *et al*, 2000). Ruiz *et al*. (1997) reported sludge flotation and significant decrease in total COD removal efficiency down to 59% at OLRs of 6.5 kg  $\text{COD/m}^3/\text{d}$  from the UASB reactor. The total COD removal efficiency in the AF was also dropped to less than 50% at an OLR higher than 6 kg  $\text{COD/m}^3/\text{d}$ . An anaerobic fluidizedbed reactor treating slaughterhouse wastewater achieved 75 % COD reduction at an OLR of 54.0 kg COD/m<sup>3</sup> /d (Borja *et al*., 1995). The feasibility of the ASBRs was demonstrated in laboratory reactors at a temperature of 30ºC treating slaughterhouse wastewater. 90 to 96 % COD removal was achieved at OLRs from 2.07 to 4.93 kg COD/m<sup>3</sup>/d (Massé *et al.*, 2000). The treatment of slaughterhouse wastewater was also carried out in the two pilot-scale SGBR systems (Park et al, 2012; Roth and Ellis, 2004).



The SGBR is a recently developed high rate anaerobic system. The key design feature of the SGBR is higher biomass concentration since a deep bed of active granules is utilized, resulting in increased treatment efficiency. Besides high COD removal efficiency, operational simplicity and lower capital and operating costs are also advantages of the SGBR. The feasibility of the reactor has been demonstrated in a number of laboratory and pilot studies on wastewater treatment including municipal wastewater, landfill leachate, and non-fat dry milk (Debik *et al*., 2005; Evans and Ellis, 2005; Mach and Ellis, 2000).

A number of models have been developed to describe the kinetics of substrate utilization for anaerobic treatment processes. The Stover-Kincannon model and the Grau second-order model are the most widely used mathematical models for determining kinetic coefficients. These models have been applied in studies on the treatment of food processing wastewater using the anaerobic contact reactor, soybean processing, papermill, simulated starch wastewater with the anaerobic filter, winery wastewater with the anaerobic fixed bed reactor, and textile and municipal wastewater using the UASB (Ahn *et al*., 2000; E.Senturk *et al*., 2010; Isik *et al*., 2005; Rangaraj *et al*., 2009; Yilmaz *et al*., 2008; Yu *et al*., 1998). However, kinetic models of the SGBR for wastewater treatment from hog slaughterhouses have not been investigated. Therefore, the objective of this study was to determine the kinetics of the two pilot-scale SGBR systems (hereafter referred to as R1 and R2) and to compare kinetic models applied for describing the substrate utilization of the SGBR treating slaughterhouse wastewater. In order to determine kinetic coefficients, mathematical models including the Grau second-order model and the Stover-Kincannon model were applied to the SGBR.



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### **Materials and methods**

#### *Wastewater source and characteristics*

Wastewaters generated from the meat processing plants were pretreated by the dissolved air floatation (DAF) system in the plant, and then pumped into feed tank for storage of influent wastewater due to the hourly and daily fluctuations in wastewater discharge quantity and quality. The average pH levels were neutral or slightly acidic. Chemicals such as sodium hydroxide (NaOH) and sodium bicarbonate (NaHCO<sub>3</sub>) were not added to the influent for pH adjustment. The average  $BOD<sub>5</sub>$  and COD values showed that meat processing wastewaters had a relatively high organic strength. In addition, the wide ranges for  $BOD<sub>5</sub>$  and  $COD$  concentrations of wastewaters reflected that daily, weekly, and seasonal variations in discharge quality from the plant. Slaughterhouse wastewater also contained high concentrations of suspended solids (SS), originating from pieces of fat, grease, hair, flesh, manure, and undigested feed (Bull *et al*., 1982). The  $BOD<sub>5</sub>/COD$  ratio was used for the determination of the biodegradability of the organic compounds in slaughterhouse wastewater. The ratio between 0.4 and 0.8 is considered to be readily biodegradable (Metcalf and Eddy, Inc., 1991). The observed ratios were greater than more than 0.4, with mean values of 0.49 and 0.73 for R1 and R2, respectively, which indicated that most of the organic compounds in these wastewaters were fairy biodegradable. The characteristics of slaughterhouse wastewater are given in Table 4−1.



Parameter	Value		
	R <sub>1</sub>	R <sub>2</sub>	
pH	$6.90 \pm 0.44$	$5.64 \pm 0.26$	
Alkalinity, mg/L as $CaCO3$	$630 \pm 107$	$264 \pm 157$	
TSS, mg/L	$840 \pm 491$	$2,355 \pm 1,321$	
VSS, mg/L	$704 \pm 431$	$2,255 \pm 1,319$	
Total COD, mg/L	$3,137 \pm 814$	$7,864 \pm 4,294$	
Soluble COD, mg/L	$1,749 \pm 368$	$3,489 \pm 985$	
$BOD_5$ , mg/L	$1,543 \pm 202$	$5,732 \pm 1,522$	
$VFA$ , mg/L as $HAc$	$486 \pm 159$	$936 \pm 385$	

**Table 4**−**1. Characteristics of slaughterhouse wastewater**

#### *Reactor set-up and operation*

The two pilot-scale SGBR systems fabricated with polypropylene were installed at meat processing plants in Austin, Minnesota and Denison, Iowa. The pilot-scale SGBR systems consisted of a 1000-gallon reactor with different working volumes (700 and 500 gallons for R1 and R2, respectively), storage tanks for influent and effluent,  $\frac{3}{4}$ -inch PVC piping and fittings, a ChronTrol controller/timer, Masterflex peristaltic pumps, and a gas meter. The anaerobic granules were obtained from an operating UASB at City Brewing Company in La Crosse, Wisconsin. R1 and R2 reactors were seeded with approximately 650 and 400 gallons of anaerobic granules, respectively. The anaerobic granules were transferred using a progressive cavity pump to avoid the disintegration of the granules. The meat processing wastewater was pumped into a feed tank from the DAF for storage of influent wastewater. Feed tanks were installed to compensate for fluctuations in wastewater pH and organic strength. The wastewater from feed tank was then fed into the SGBR using peristaltic pump. The influent wastewater was evenly distributed over the granular bed using perforated distribution pipes located in the headspace of the reactor. Underdrain system consisted of perforated  $\frac{3}{4}$ -inch PVC pipes within the graded gravel layer installed along the bottom of the reactor, designed to provide uniform



collection of the treated effluent. The backwash water using collected effluent was also uniformly distributed throughout the granular bed by the underdrain system. The biogas produced by the system was passed through the gas scrubber filled with a mixture of coarse and fine steel wool to remove hydrogen sulfide  $(H<sub>2</sub>S)$  and measured using wet-test gas meters (Schlumberger Industries, Dordrecht, The Netherlands). The pressure and water level changes inside the reactor were monitored with the attached manometer and side mounted tubular level indicator, respectively. The liquid level was maintained at working volume of each reactor by using an adjustable effluent overflow pipe. R1 and R2 reactors were continuously operated at the average OLRs of 1.09 and 1.41 kg  $\text{COD/m}^3/\text{d}$ , respectively, during the start-up period. After the acclimation period, the average organic loading rates for R1 and R2 were increased stepwise to 2.91 and 6.19 kg  $\text{COD/m}^3/\text{d}$  by shortening the HRTs stepwise from 48 to 28 and 20 h, respectively.

#### *Data collection and analytical methods*

The parameters including chemical oxygen demand (COD), soluble chemical oxygen demand (SCOD), volatile fatty acids (VFAs), biochemical oxygen demand (BOD), total suspended solids (TSS), and volatile suspended solids (VSS) were determined in accordance with Standard Methods for the Examination of Water and Wastewater (APHA,1998). The influent and effluent wastewater pH were measured using an electronic pH meter (Thermo Orion 210A). 24-hour composite influent and effluent samples were collected from storage tanks for analysis. The biogas was measured with wet-test gas meters, and collected with 100-mL glass gas sampling tube. The biogas composition was analyzed by the laboratory in the meat processing plant and ISU analytical laboratory using a Gow Mac gas chromatograph. Hydrogen



sulfide  $(H<sub>2</sub>S)$  measurement was performed on-site using a Dräger accuro gas detector pump with  $H<sub>2</sub>S$  detector tubes.

## **Results and discussion**

#### *Performance of the SGBR systems*

Influent COD concentration and COD removal rates under various organic and hydraulic loading conditions were summarized in Table 4−2. During start-up period, the COD removal efficiencies of 94 and 92% were observed in R1 and R2 at the initial OLR of 1.09 and 1.41 kg COD/m<sup>3</sup>/d, respectively. The COD removal rate in R2 at OLR of 1 kg COD/m<sup>3</sup>/d was significantly improved as the system stabilized. Both SGBR reactors achieve high organic removal rates within a very short start-up period (21 days for R1 and 25 days for R2) since the anaerobic granules obtained from an operating UASB were used as seed granules. The average OLR applied to R1 and R2 were increased stepwise from 1.09 to 2.91 and from 1.41 to 6.19 kg  $\text{COD/m}^3$ /d, respectively, by shortening the HRT.



Reactor	Day	$\tilde{\phantom{a}}$ HRT(h)	$\mathrm{COD}_{\mathrm{Inf}}\,(\mathrm{mg}/\mathrm{L})$	OLR (kg $\text{COD/m}^3/d$ )	COD removal $(\% )$
R <sub>1</sub>	$1 - 8$	48	$2179 \pm 94$	1.09	$93.4 \pm 0.3$
	$9 - 43$	40	$2533 \pm 450$	1.52	$94.0 \pm 0.8$
	44-64	36	$3225 \pm 456$	2.15	$94.9 \pm 0.9$
	65-97	32	$3728 \pm 517$	2.80	$94.4 \pm 0.8$
	100-128	28	$3395 \pm 590$	2.91	$93.5 \pm 1.2$
Average			$3137 \pm 711$	2.25	$94.1 \pm 1.0$
R <sub>2</sub>	$1 - 30$	96	$5659 \pm 1753$	1.14	$92.1 \pm 5.8$
	$31 - 62$	48	$6773 \pm 1722$	3.39	$95.6 \pm 2.1$
	63-132	36	$9238 \pm 3141$	5.52	$96.6 \pm 1.4$
	133-174	30	$8494 \pm 2598$	6.00	$96.0 \pm 1.5$
	177-216	24	$6556 \pm 1899$	5.47	$95.7 \pm 1.8$
	217-265	20	$6710 \pm 1907$	6.19	$95.4 \pm 2.0$
Average			$7864 \pm 4294$	4.84	$95.4 \pm 2.9$

**Table 4−2. Performance of two pilot scale SGBR systems treating slaughterhouse wastewater under steady state condition**

The effect of the organic loading rate on the process performance was evaluated based on the COD removal efficiency in the SGBR systems with different OLR (Figure 4−1). R1 and R2 attained the average COD removal rates of 94 and 95% at OLR ranging from 1.01 to 3.56 and 0.94 to 12.76 kg COD/m<sup>3</sup>/d, respectively. The variation of organic loading rates for R2 was due to high fluctuation of COD concentrations from the DAF unit ranging from 2720 to 15950 mg COD/L. Both SGBR reactors could cope with hydraulic overloading by reducing the HRT and organic shock loads caused by sudden increase in waste strength. In addition, high organic removal efficiencies were maintained even at the maximum organic loading rate applied to each system. The average values of COD removal efficiency from both SGBR reactors were not decreased with increase in loading rates.





**Figure 4−1. COD removal efficiency in the SGBR systems with different OLR**

*Monitoring parameters and the stability of the SGBR*

The pH, alkalinity, VFA, and ammonia are important parameters for monitoring and control of the anaerobic microbial treatment process. As presented in Table 4−3, the average effluent pH, alkalinity, and VFA were 7.49, 1,158 mg/L as  $CaCO<sub>3</sub>$ , and 21 mg/L as HAc for R1 and 7.27, 715 mg/L as  $CaCO<sub>3</sub>$ , and 18 mg/L as HAc for R2, respectively. The pH values of the influent wastewater have varied from 6.1 to 7.9 for R1 and from 4.8 to 6.3 for R2, respectively. The pH values of the effluent were maintained in the optimal range (6.5 to 8.2) for the methanogenic microorganisms (Speece, 1996). In addition, the ratio of VFA to alkalinity, indicating process stability, was also monitored (Ripley *et al*., 1986). A VFA to alkalinity ratio


less than 0.3 reflects stable operating conditions, while a ratio between 0.3 and 0.4 indicates a potential for upset and possible need for corrective action. When the ratio exceeds 0.8, methanogens can be inhibited by VFA accumulation and the digester becomes acidified (WPCF Manual of Practice No. 16, 1987). Both SGBR reactors were operated at VFA/alkalinity ratio less than 0.03 on average as shown in Figure 4−2. These lower ratios were attributed to low effluent VFA concentrations and the increase in effluent alkalinity observed in both reactors. It could have resulted from favorable conditions for the methanogenic microbes and the generation of bicarbonate from the conversion of protein to ammonia during the operation. Ammonia–N released by the destruction of protein reacts with carbon dioxide produced by the biochemical reaction to produce ammonium bicarbonate. This effect contributed sufficient buffering capacity in the SGBR system to tolerate pH variations so that pH adjustments were not necessary. This fact may reduce operating costs during a full-scale anaerobic treatment of the slaughterhouse wastewater. A pH in the normal range and low VFA/alkalinity ratio indicate that the anaerobic microorganisms were operating in a stable condition without accumulation of fermentation intermediates such as VFAs.

Reactor	Day	HRT(h)	pH	VFA (mg/L as HAc)	Alkalinity (mg/L as $CaCO3$ )
R1	$1-8$	48	$7.59 \pm 0.15$	$21 \pm 4$	$1,084 \pm 116$
	$9 - 43$	40	$7.78 \pm 0.29$	$20 \pm 5$	$1,156 \pm 120$
	44-64	36	$7.49 \pm 0.25$	$22 \pm 5$	$1,114 \pm 265$
	65-97	32	$7.31 \pm 0.14$	$20 \pm 2$	$1,139 \pm 59$
	100-128	28	$7.32 \pm 0.19$	$21 \pm 6$	$1,233 \pm 74$
Average			$7.49 \pm 0.29$	$21 \pm 5$	$1,158 \pm 142$
R <sub>2</sub>	$1-30$	96	$6.88 \pm 0.19$	$16 \pm 4$	$613 \pm 43$
	$31-62$	48	$7.19 \pm 0.3$	$13 \pm 2$	$516 \pm 80$
	63-132	36	$7.44 \pm 0.29$	$19 \pm 7$	$786 \pm 114$
	133-174	30	$7.36 \pm 0.19$	$19 \pm 6$	$758 \pm 125$
	177-216	24	$7.25 \pm 0.2$	$21 \pm 10$	$718 \pm 103$
	217-265	20	$7.18 \pm 0.23$	$19 \pm 4$	$613 \pm 43$
Average			$7.27 \pm 0.28$	$18 \pm 6$	$715 \pm 132$

**Table 4**−**3. Variation of pH, VFA, and alkalinity of the two pilot scale SGBR systems**





#### *Conventional Monod kinetics*

The rate of change of biomass in the reactor depends on the influent and effluent biomass and the biomass growth and decay in the system.

$$
\frac{\mathrm{d}X}{\mathrm{d}t} = \frac{Q}{V}\left(X_0 - X_E\right) + \left(\mu - K_d\right)X\tag{4.1}
$$

where  $X =$  concentration of microorganisms, g VSS/L



 $Q =$  flow rate of influent, L/day

 $V =$  reactor volume, L

 $X_0$  and  $X_E$  = microorganisms in influent and effluent, g VSS/L

 $\mu$  = specific growth rate, 1/day

 $K_d$  = endogenous decay coefficient,  $1/day$ 

The solids retention time (SRT),  $\theta_C$  is defined as the average time of the retained biomass in the system, which is also called as mean cell residence time (MCRT). It is the ratio of the total biomass in the reactor to the biomass in the effluent and wasted biomass from the system during the backwash procedure in a given time period as given below:

$$
\theta_c = \frac{VX}{QX_E} \tag{4.2}
$$

The calculated average SRT in R1 and R2 were 243 and 157 days, respectively. There was a trend of decreasing SRT with decrease in HRT in both SGBR systems. Evans (2004) also reported that the SRT in the SGBR was much higher at 15ºC than at 8ºC at the same HRT, and the SRT increased with increasing HRT.

The relationships between the specific growth rate of the microorganisms and the concentration of the limiting substrate for growth were described by the Monod equation:



$$
\mu = \mu_{\text{max}} \frac{S}{K_S + S} \tag{4.3}
$$

where  $\mu_{max}$  = maximum specific growth rate, 1/day

 $K_S$  = half velocity constant, mg/L

Assuming biomass concentrations are at steady state  $\left(\frac{dX}{dt} = 0\right)$  and microorganisms in the influent are negligible, Eq. (4.1) can be simplified as follows:

$$
\frac{Q}{V}X_E = (\mu - K_d)X\tag{4.4}
$$

$$
\mu = \frac{QX_E}{VX} + K_d \tag{4.5}
$$

$$
\mu = \frac{1}{\theta_c} + K_d \tag{4.6}
$$

$$
\mu_{\text{max}} \frac{S}{K_s + S} = \frac{1}{\theta_c} + K_d \tag{4.7}
$$

Eq. (4.8) can be obtained from Eq. (4.7) to predict the effluent concentration under steady-state conditions as follows:



$$
S = \frac{K_s (K_d + \frac{1}{\theta_C})}{\mu_{\text{max}} - K_d - \frac{1}{\theta_C}}
$$
(4.8)

The rate of change of substrate concentration in the system can be described by

$$
-\frac{\mathrm{d}S}{\mathrm{d}t} = \frac{Q}{V}S_0 - \frac{Q}{V}S - \frac{\mu X}{Y}
$$
(4.9)

At steady state conditions, the accumulation term, d*S/*d*t*, reduces to zero. Eq. (4.9) can be rearranged by substituting Eq.  $(4.6)$  for  $\mu$ , as follows:

$$
\frac{(S_0 - S)}{\theta_H X} = \frac{1}{Y} \left( \frac{1}{\theta_C} + K_d \right) = \frac{1}{Y} \frac{1}{\theta_C} + \frac{K_d}{Y}
$$
\n(4.10)

The values of  $Y$  and  $K_d$  can be determined from the slope and intercept of equation of the straight line by plotting Eq. (4.10). Eq. (4.7) can be rearranged to obtain values of  $\mu_{\text{max}}$  and  $K_S$  as shown below:

$$
\frac{\theta_C}{1 + \theta_C K_d} = \frac{K_s}{\mu_{\text{max}}} \frac{1}{S} + \frac{1}{\mu_{\text{max}}} \tag{4.11}
$$





**Figure 4−3. Monod kinetic application for** *Y* **and** *K***<sup>d</sup>**

The growth yield coefficient, *Y*, determined from the slope was 0.10 and 0.09 g VSS/g COD for R1 and R2, respectively (Figure 4−3). The values of biomass yield indicates overall yield for the mixed culture of acidogens (0.14-0.17 g VSS/g COD) and acetoclastic methanogens (0.01-0.05 g VSS/g COD). The estimated decay coefficient for R1 and R2 were  $3.56 \times 10^{-4}$  day<sup>-1</sup> and  $8.27 \times 10^{-4}$  day<sup>-1</sup>, respectively. Yoochatchaval *et al.* (2008) also have reported that the growth yield of retained sludge (0.13 g VSS/g COD) and very low decay constant of  $1.0 \times 10^{-4}$  day<sup>-1</sup> from the EGSB reactor treating low strength wastewater at 20ºC.





**Figure 4–4.** Monod kinetic application for  $\mu_{\text{max}}$  and  $K_{\text{S}}$ 

Obtained values of  $\mu_{\text{max}}$  and  $K_S$  for R1 were 0.011 day<sup>-1</sup> and 257 mg COD/L, respectively. However, Monod kinetics could not describe the performance of R2.

*Grau second order model for SGBR*

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The general equation of the Grau second order kinetic model is as follows:

$$
\frac{dS}{dt} = -k_s X \left(\frac{S_e}{S_0}\right)^2 \tag{4.12}
$$

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where  $k_s$  is the substrate removal rate constant (1/d), *X* is the average biomass concentration in the reactor (mg VSS/L),  $S_e$  and  $S_0$  are the effluent and influent substrate concentration (mg COD/L), respectively. Eq. (4.12) can be integrated and then linearized as follows:

$$
\frac{S_0 \theta_H}{S_0 - S_e} = \theta_H + \frac{S_0}{k_s X} \tag{4.13}
$$

If the second term on the right side of the equation is assumed to be constant, Eq. (4.13) can be written as follows:

$$
\frac{S_0 \theta_H}{S_0 - S_e} = b\theta_H + a \tag{4.14}
$$

$$
\frac{\theta_{H}}{E} = b\theta_{H} + a \tag{4.15}
$$

where (*S0−Se*)/*S<sup>0</sup>* is the substrate removal efficiency and symbolized with *E*. In order to determine the second-order substrate removal rate constant  $k<sub>S</sub>$ , *a* and *b*, Eq. (4.15) can be plotted (Figure. 4−4).





**Figure 4−5. Second-order kinetic model application**

The kinetic parameters, *a* and *b*, can be calculated from the intercept and slope of the straight line, respectively. Calculated values of *a* and *b* were found as 0.017 and 1.05 for R1, and 0.0045 and 1.0396 for R2, respectively, with a high correlation coefficient ( $\mathbb{R}^2$  > 0.99). Assuming that the average concentration of biomass in the SGBR was 24,000 mg/L, the second-order substrate removal rate constants can be obtained from value *a*. Estimated values of *a* and *b* can be used to predicting effluent concentrations. Eq. (4.14) can be written as below:

$$
S = S_0 \left( 1 - \frac{\theta}{0.017 + 1.05\theta} \right) \quad \text{for} \quad \text{R1}
$$
\n
$$
\tag{4.16}
$$

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$$
S = S_0 \left( 1 - \frac{\theta}{0.0045 + 1.0396\theta} \right) \text{ for R2}
$$
 (4.17)

Predicted COD concentrations were calculated by using Eq. (4.16) and (4.17) based on Grau second-order kinetic model. Figure 4−6 shows the relationship between the measured and predicted COD concentrations. The predicted values were consistent with the experimental data in R1 compared to R2. This was possibly due to the highly fluctuating influent COD levels in R2. Observed COD removal rates were 94% in R1 and 95% in R2. Equation for predicted COD in R1 and R2 estimated 94% and 96% removal efficiency in R1 and R2, respectively.



**Figure 4−6. Observed and predicted COD concentrations for Grau second order model**



The second-order substrate removal rate constant  $k_2(a=S_0/k_2X)$  were 3.8 day<sup>-1</sup> for R1 and 34 day<sup>-1</sup> for R2. Higher value of second-order substrate removal rate constant in R2 was in the similar range with values for UASBR treating young landfill leachate (Ozturk et al., 1998).

#### *Modified Stover–Kincannon model for SGBR*

The Stover-Kincannon model, originally proposed for rotating biological contactors (RBCs), assumed that the substrate utilization rate could be expressed as a function of the organic loading rate for biofilm reactors. The contribution of the suspended biomass to substrate removal was assumed to be negligible in comparison to the attached biomass on the support media. Therefore, the disc surface area of the rotating biological contactor was used to represent the total attached-growth active biomass concentration in the original model (Kincannon, 1982). However, the suspended microorganisms within the media interstitial void spaces between the packing and biogrowth was considered to be a significant factor in substrate removal in anaerobic filters (Song and Young, 1986, Tay *et al*., 1996) and the effective volume of the reactor can be used instead of the surface area of the support media (Yu HQ *et al*., 1998). Therefore, at steady state, the modified Stover–Kincannon model can be expressed as follows:

$$
\frac{dS}{dt} = \frac{U_{\text{max}}(QS_0/V)}{K_{\text{B}} + (QS_0/V)}
$$
(4.18)

where d*S*/d*t* is defined as follows:

$$
\frac{\mathrm{d}S}{\mathrm{d}t} = \frac{Q}{V} \left( S_0 - S_e \right) \tag{4.19}
$$

where  $dS/dt$  is the substrate utilization rate (g/L/d),  $U_{\text{max}}$  is the maximum removal rate constant (g/L/d),  $K_B$  is the saturation value constant (g/L/d), Q is the flow rate (L/d), V is the working



volume of the reactor (L), and  $S_0$  is the influent substrate concentration (g TCOD/L), and  $S_e$  is the effluent substrate concentration (g TCOD/L). Eq. (4.20) can be obtained from the linearization of the inverse of Eq. (4.18) and Eq. (4.19):

$$
\left(\frac{dS}{dt}\right)^{-1} = \frac{V}{Q(S_0 - S_e)} = \frac{K_B}{U_{\text{max}}} \frac{V}{QS_0} + \frac{1}{U_{\text{max}}}
$$
(4.20)

If the inverse of the substrate utilization rate is plotted against the inverse of the total loading rate, the linear relationship can be obtained as shown in Figure 4**−**7.



**Figure 4−7. Modified Stover-Kincannon model application**



The values of  $U_{\text{max}}$  and  $K_{\text{B}}$  were obtained from the slope and intercept of Eq. (4.20). According to Figure 4-7, the predicted values of  $U_{\text{max}}$  and  $K_{\text{B}}$  were 192.3 g COD/L/d and 206.6 g COD/L/d for R1, and 243.9 g COD/L/d and 259.5 g COD/L/d for R2, respectively. The predicted values of  $U_{\text{max}}$  were significantly higher than the maximum OLR (3.56 and 12.76 g COD/L/d for R1 and R2, respectively) applied to the system during the study, indicating the potential for the SGBR to deal with high strength slaughterhouse wastewater. A mass balance of substrate is expressed as follows:

$$
QS_0 = QS_e + V\left(\frac{dS}{dt}\right) \tag{4.21}
$$

By combining Eq. (4.18) and (4.21), the effluent substrate concentration can be obtained as follows:

$$
QS_0 = QS_e + V \left( \frac{U_{\text{max}} (QS_0 / V)}{K_{\text{B}} + (QS_0 / V)} \right)
$$
(4.22)

$$
S_e = S_0 - \frac{U_{\text{max}} S_0}{K_{\text{B}} + (Q S_0 / V)}
$$
(4.23)





**Figure 4−8. Observed and predicted COD concentrations for Modified Stover-Kincannon model**

Figure 4−7 shows the relationship between the observed and predicted effluent COD concentration. However, predicted values were usually higher than experimental values. This may be due to the entrapment of particulate COD within the SGBR.



<b>Substrates</b>	Type of reactor	Temperature (°C)	$U_{\text{max}}$ $(g \text{ COD/L/d})$	$K_{B}$ $(g \text{ COD/L/d})$	References
Slaughterhouse	SGBR(R1)	$22 \pm 3$	192.3	206.6	This study
Slaughterhouse	SGBR(R2)	$20 \pm 3$	243.9	259.5	This study
Poultry Slaughterhouse	<b>SGBR</b>	22	164.48	177.21	E. Debik, 2009
Food Processing	Anaerobic contact reactor	$35 \pm 2$	22.925	23.586	E.Senturk, 2010
Milk permeate	Anaerobic moving bed biofilm reactor	35	89.3	102.3	Wang, 2009
Simulated textile wastewater	<b>UASB</b>	30	7.5	8.2	Isik & sponza, 2005
Simulated starch	Anaerobic Filter	35	49.8	50.6	Ann & Foster, <b>2000</b>
Simulated starch	Anaerobic Filter	55	667	702	Ann & Foster, 2000
Soybean processing	Anaerobic Filter	$35 \pm 1$	83.3	85.5	Yu, 1998

**Table 4**−**4. Comparison of the kinetic coefficients** 

The kinetic coefficients obtained in the current study were compared with those obtained from other anaerobic processes for the various substrates (Table 4−4). Although these values were estimated from various reactor configurations, wastewater characteristics, and operating conditions, higher values were obtained from SGBR systems. The thermophilic reactors treating simulated starch and paper mill wastewater had a significantly higher maximum utilization rate than the mesophilic reactors (Ahn and Forster, 2000; Yilmaz *et al*., 2008). These results showed that the SGBR systems under ambient conditions achieved similar or even higher maximum utilization rates while other anaerobic processes were operated under mesophilic condition. In terms of maximum utilization rate, the SGBR systems were not significantly affected by low temperatures.

## **Conclusions**

The two pilot-scale SGBR exhibited excellent process performance for the treatment of slaughterhouse wastewater. R1 and R2 attained the average COD removal rates of 94% and 95%



at OLR ranging from 1.01 to 3.56 and 0.94 to 12.76 kg  $\text{COD/m}^3/\text{d}$ , respectively.

During the operation of reactors, the solid retention times of 243 and 157 days for the R1 and R2, respectively were obtained. Henze (2008) suggested that the minimum SRT should always three times longer than the doubling time of the microorganisms. *Methanosaeta* typically has a doubling time of 4-9 days while *Methanosarcina* has shorter doubling times (1-2 days) (Zinder, 1988). Therefore, long SRT enabled slow growing methanogens to get sufficient time to grow and stabilize, and promoted the proliferation of methanogenic bacteria in the granular sludge.

It was shown that Monod kinetics is not very appropriate for describing the performance of the SGBR for treating slaughterhouse wastewater since Monod kinetics was demonstrated using pure cultures and simple substrates. Digestion of complex organic matters could result in deviation from the Monod relationship in the SGBR. Only the hydrolyzed compounds may be considered as the growth-limiting substrate in terms of the Monod kinetics. A significant correlation was also not found between predicted and measured COD concentrations for Grau second-order kinetic model and modified Stover-Kincannon model since high COD removal efficiencies were maintained regardless of organic loading rates. Predicted values by modified Stover-Kincannon model were usually higher than experimental values. This may be due to the entrapment of particulate COD within the SGBR.



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# **CHAPTER 5. SEPTIC WASTEWATER TREATMENT USING RECYCLED RUBBER PARTICLES (RRP) AS BIOFILTRATION MEDIA**

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## **Introduction**

Onsite wastewater treatment systems, commonly known as septic systems, are the most widely used systems in suburban and rural areas where public sewer systems are not available to handle household wastewater. Approximately one quarter of the population in the United States is served by onsite wastewater treatment systems. The most common onsite treatment system is the septic tank and soil absorption system also known as the drainfield or leach field.

The main functions of a septic tank are to separate solids from the wastewater, provide anaerobic digestion of organic matter, and provide storage for the sludge and scum. The septic tank allows the heavy solids to settle on the bottom, forming a sludge layer, and the grease and fatty solids to float to the top, forming a scum layer. Performance of septic tanks depends on the characteristics of influent, design, operation, and maintenance of the septic tank. Typical septic tank removal efficiencies have been reported as follows: biochemical oxygen demand (BOD5) 31-68%, total suspended solids (TSS) 30-81%, fecal coliform 25-66% (Boyer and Rock, 1992; Rahman *et al*., 1999; Rock and Boyer, 1995; Seabloom *et al*., 1982;). However, septic tank effluent (STE) still contains disease-causing pathogens and excessive nutrients such as nitrogen and phosphorus. Therefore, effluent flows from the septic tank outlet to a subsurface wastewater



infiltration system (SWIS) that includes soil, sand, or other media for further treatment through biological processes by microorganisms, chemical adsorption, and physical filtration. Approximately one-third of the land area in the United States is suitable for conventional soil absorption systems. Alternative septic systems can be used for the sites where an existing septic system has failed or site conditions, such as high groundwater table or small lot size, are not suitable for the installation of conventional septic systems.

Filtration systems are one of the most widely used alternative septic systems. Several types of permeable material, including sand, gravel, peat, and synthetic materials such as textile, glass, or foam, have been used as the filter media. As septic tank effluent is distributed across the top of the media and passes through the filter, most of the suspended solids are filtered and dissolved organic compounds are removed by adsorption and biodegradation within biofilms developed from the growth of microorganisms on the surface of the media. Sand filters are the most common type of media filtration system used in conjunction with septic systems. If the system is hydraulically overloaded, the accumulation of excessive biomass or entrapped organic matter due to decreased rates of decomposition can occupy the pore space, resulting in filter clogging and surface ponding. Organic filter media such as peat may decompose and degrade over time, thus requiring periodic replacement.

Recycled rubber has been used in various applications, including asphalt, rubber mulch, and aggregate substitute in drainage systems for landfills and septic systems. Several studies have concluded that the effects of tire derived aggregate on water quality were negligible and the concentrations of contaminants leaching from scrap tires such as Fe, Mn, Zn and Al were



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below the limits set by drinking water standards (Downs *et al*., 1996; Edil and Bosscher, 1992; Humphrey *et al*., 1997; Humhrey and Katz, 1996; Lerner *et al*, 1993; O'Shaughnessy and Garga, 2000; Sengupta and Miller, 2000). Recycled rubber has also been used as packing media in trickling filters for landfill leachate treatment and, biofilter media for volatile organic compounds and odor removal (Mondal and Warith, 2008; Park *et al*., 2011). Recycled rubber particles (RRP) can be used as filter media or substitutions for gravel in septic system drainfields due to the high surface area for attached growth of biofilm as well as economic benefits. Therefore, in this study biofilter systems using three different filter media, including RRP, peat, and gravel, were demonstrated at laboratory scale for treating septic tank effluent and the treatment performance of a recycled rubber particles system was compared to a conventional gravel system and a peat moss system.

## **Materials and methods**

#### *Laboratory-scale biofilter reactors configuration and operation*

Three identical laboratory-scale columns packed with different types of media (RRP, peat, and gravel) for treating septic tank effluent were operated in single pass modes to evaluate the performance of three different filter media. A schematic of the biofilters is shown in Figure 5−1. Each biofilter made of Plexiglass had a width of 0.5 ft and an overall height of 3 ft. Each column was filled with biofilter media to provide a total bed depth of 2.7 ft and a total bed volume of approximately 5 gallons. Pea gravel layers, approximately 4 inches, were placed at the bottom of both RRP and peat columns to support filter media and prevent the underdrain from clogging. Septic tank effluent was fed intermittently into biofilters by a timer-controlled pump,



and then distributed evenly over the surface of the media through polyethylene bottles with perforated bottom. The reactors were operated at room temperature. Septic tank effluent can vary in quality depending on the characteristics of the wastewater and condition of the tank. The septic tank effluent used in this study was collected from residential area, having higher suspended solids and organic matter concentrations in comparison to typical septic tank effluent as presented in Table 5−1 (Bounds, 1997; Crites and Tchobanoglous, 1997; Long, 1997; Otis *et al*., 1973; Seabloom *et al*., 1982).

Parameter		Average concentration (mg/L)			
	Influent	<b>Typical STE</b>			
<b>TSS</b>	$401 \pm 456$	44-118			
VSS	$341 \pm 379$	N/A			
<b>COD</b>	$468 \pm 348$	228-338			
BOD <sub>5</sub>	$204 \pm 81$	85-190			
$NH_{3}-N$	$58.2 \pm 18.8$	$30 - 50$			

**Table 5−1. Characteristics of septic tank effluent**

Each biofilter was rinsed intermittently with tap water prior to the start of the experiment for 7 days to remove any impurities and minimize the potential interference in chemical oxygen demand (COD) determination, which could be caused by organic matter leaching from biofilter media. For enhanced biofilm formation during the start-up period, each biofilter was seeded with 5 L of activated sludge (1.0 g/L VSS) from the Boone Water Pollution Control Plant, and the hydraulic loading rate (HLR) was maintained at 1.4 gallon per day per square foot (gpd/ft<sup>2</sup>).





**Figure 5−1. Schematic of biofilter systems**

### *Analytical procedures*

Influent and effluent samples were analyzed three times a week to monitor the performance of the reactor. The water quality parameters including chemical oxygen demand (COD), biochemical oxygen demand (BOD), total suspended solids (TSS), and volatile suspended solids (VSS), were measured in accordance with Standard Methods (APHA, 1998). COD was measured using the Closed Reflux Titrimetric Method (Standard Methods, section 5220 C). TSS and VSS were analyzed by the filtration method (Standard Methods, section 2540 D and E) with glass fiber filter paper (Whatman GF/C, 1.2  $\mu$ m). Ammonia nitrogen (NH<sub>3</sub>-N) was measured according to the ammonia-selective electrode method (Standard Methods, section 4500 D and E). Nitrate nitrogen  $(NO<sub>3</sub>-N)$  was determined by cadmium reduction method using a HACH DR 3000 spectrophotometer. Fecal coli form was determined using A-1 medium test kit from HACH.



*Ammonia adsorption batch test* 

Batch adsorption tests were conducted to evaluate the ability of RRP to adsorb ammonia. Various RRP dosages ranging from 0.2 to 10g were added into Erlenmeyer flasks filled with 150mL of ammonium chloride solution with a fixed concentration of  $10$ mg/L NH<sub>4</sub><sup>+</sup>. Each mixture was shaken for 5 min using an automatic shaker (Incubator shaker series 2, New Brunswick Scientific Co., Inc.) at 180 rpm, and then allowed to settle for 5 min. Supernatant solutions were analyzed for ammonia concentration. Amounts of ammonia adsorbed by RRP were calculated as the difference between amounts of ammonia initially added and those remaining in the supernatant solutions. The ability of RRP to adsorb ammonia was assessed using Freundlich and Langmuir isotherms.

## **Results and discussion**

#### *Start-up period of laboratory-scale biofilter reactors*

Significant higher concentrations of COD were observed in the effluent from peat and RRP biofilters during the initial operation period as shown in Figure 5**−**2. Therefore, each biofilter was flushed with tap water prior to the start of the experiment to wash off any impurities and prevent interferences in analytical measurements. Colored effluent containing small peat particles was released from the peat media. Peat consists primarily of organic matter and it leaches colored organic matter such as humic and fulvic acids. These leachates may contribute to the effluent COD and lower apparent treatment efficiency. Rock *et al*. (1984) concluded that the relatively lower COD reduction rates resulted from the organic matter leached from the peat itself. Viraraghavan and Ayyaswami (1988) and Viraraghavan and Rana (1991) also reported COD contribution by the peat itself. In the RRP filter, the increase in COD concentrations could



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possibly be attributed to leaching of dissolved organic compounds such as benzothiazole derivatives, and some particulate organic matter from the RRP.



**Figure 5−2. COD concentrations in flush water**

The effluent COD concentration decreased to 165 and 220 mg/L in RRP and peat media, respectively, during the flushing period. Although the effluent COD concentrations in both systems were improved, different leaching patterns were observed during this period. Since the water was unable to penetrate into the rubber, releases of easily leachable compounds would occur predominantly at the surface of the rubber over a relatively short period of time. The rate of leaching significantly decreased with the number of washes and exposure time over the first four days. This finding is consistent with previous studies reporting the decrease in leaching rate of dissolved organic carbon with time (Abernethy *et al*., 1996; O'Shaughnessy and Garga., 2000). The effluent COD in peat biofilter was stabilized after three days in the concentration range of about 220 to 370 mg/L, whereas the leach rate for organic matter from RRP continued to decrease at the end of this period.

After the period for prewash procedures, the biofilters were fed with septic tank effluent



at the hydraulic loading rate of 1.4 gpd/ft<sup>2</sup> during the start-up period. As shown in Figure 5–3, the effluent COD from the gravel biofilter remained stable at a low level (on average 42 mg/L). On the other hand, the effluent COD from peat was still relatively high and dramatically increased due primarily to the release of dissolved organic matter from the filter media, which were generally considered to be refractory. These compounds may in turn affect COD values in the effluent. The color of the effluent also gradually changed from light brown to dark brown with time. The relatively higher COD/BOD ratio of 25 in peat biofilter effluent during the startup period, suggested that it contained high molecular weight humic and fulvic-like compounds.

The effluent COD concentrations from the RRP biofilter were maintained at similar levels of influent COD. Suspended and colloidal particles in the influent are usually transported to the filter media and removed by several mechanisms including interception, sedimentation, and diffusion. On day 47, each biofilter was seeded with 5 L of activated sludge from the water pollution control plant to promote biofilm formation and to improve organic removal efficiencies. The hydraulic loading rate was maintained at  $1.4$  gpd/ft<sup>2</sup> during the start-up periods, which allowed the biomass to become acclimated to the wastewater and reactor configuration.



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**Figure 5−3. Variations in influent and effluent COD during the start-up period**

*Organic removal* 

Parameter	Average concentration $(mg/L)$				
	<b>STE</b>	<b>RRP</b>	Peat moss	Gravel	
<b>TSS</b>	$401 + 456$	$19 + 15$	$15 \pm 6$	$94 + 97$	
<b>VSS</b>	$341 + 379$	$10 + 7$	$11 + 5$	$48 + 37$	
<b>COD</b>	$468 + 348$	$107 \pm 19$	$240 \pm 150$	$50 \pm 31$	
BOD <sub>5</sub>	$204 + 81$	$16.1 + 22.1$	$19.9 + 7.5$	$5.1 \pm 2.4$	
$NH_{3}-N$	$58.2 \pm 18.8$	$9.2 \pm 12.1$	$15.4 \pm 13.2$	$2.1 \pm 5.5$	

**Table 5−2. Summary of the influent and effluent parameters** 

The applied hydraulic loading rate was increased stepwise from 1.4 to 5.0 gpd/ft<sup>2</sup>. The experiment was divided into five consecutive phases with different hydraulic loading rates.

Average influent and effluent concentrations are summarized in Table 5−2.





**Figure 5−4. Variations of COD, BOD, TSS, and VSS in septic tank effluents**





**Figure 5−5. Variations of COD, BOD, and TSS in effluents with hydraulic loading rate** 

The gravel biofilter provided effective organic removal regardless of the hydraulic or organic loading rate applied. The overall average removal efficiencies of COD and BOD were 86% and 97 %, respectively. Due to the high hydraulic conductivities of the gravel, seed



activated sludge was evenly distributed over the entire height of the gravel biofilter and thus biofilm development was noticed at the end of the start-up period. Solids gradually accumulated on the gravel surface during low hydraulic loading condition. Additionally, the low water holding capacity of gravel could not provide mechanical filtration. The gravel biofilter effluent had relatively high TSS concentrations throughout the study. Figure 5−6 shows a rapid increase to a peak value, followed by a decrease in TSS concentrations and average TSS concentrations increased from 10.1 to 170.3 mg/L during phase 1. As the hydraulic loading rate increased from 1.4 to 2.0 gpd/ft<sup>2</sup>, sloughing of biomass loosely attached on the surface of filter media or held in the void of gravel media occurred by increased hydraulic shear forces, and then sloughed biomass passed through the high void space of gravel media. As shown in Figure 5−6, low values of the VSS to TSS ratio in effluent were observed in this phase since suspended inorganic solids accumulated on the gravel surface also were carried away in the effluent. The growth and endogenous decay of biomass would also contribute to effluent VSS concentration. Therefore, the average VSS to TSS ratio in phase 1 gradually increased from 0.38 to 0.64 at the end of phase 4.



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**Figure 5−6. Variations of ratio of VSS to TSS in effluent from gravel filter** 



**Figure 5−7. COD/BOD ratio in the effluent of the peat and RRP filter**



The effluent concentrations of the peat biofilter remained relatively constant at an average BOD of 21 mg/L and TSS of 15 mg/L, which corresponds to overall removal rates of 88 and 93%, respectively (Figure 5−5). However, effluent COD levels were showing increasing trends and often exceeded those of influent until phase 1, while BOD and TSS concentrations were found to be relatively stable. Figure 5−7 shows the average COD/BOD ratios in effluent from the peat filter, which was 22 and 18 during the start-up period and phase 1, respectively. These indicated that non-biodegradable organic matters contributing soluble COD to the effluent were still leaching from the peat. In addition, these compounds have not been found to be detrimental to the treatment capabilities in terms of BOD and suspended solids. During phase 2 and 3, COD and TSS concentrations in the septic tank effluent were significantly increased. However, effluent COD decreased with time during these two phases, resulting in a COD/BOD ratio of below 10 and COD reduction of 68%. On the other hand, ponding of influent on the peat surface occurred frequently due to clogged peat media during this period. On these occasions, the peat biofilter operation was temporarily stopped. It could be that as COD and TSS concentrations in the influent and also hydraulic loading rates were increased, the pore size of peat could be reduced by several factors such as accumulation of excessive biological slime or suspended solids and decomposed or compacted peat media. Consequently, the peat biofilter may have hydraulic conductivity and poor drainage, causing accelerated clogging of the biofilter as well as limited oxygen diffusion. The operation was eventually discontinued at the end of phase 3 with a hydraulic loading rate of 4.0 gpd/ft<sup>2</sup> due to the persistent ponding.

In Figure 5−5, it can be seen that COD, BOD, and solids concentrations in the RRP biofilter dropped significantly as the biofilm began to develop and the leaching rate of organic



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compounds decreased during start-up period. Despite the increase in hydraulic loading, effluent COD concentrations continued to decrease, and thus 61, 80, 89, and 83% of removal efficiencies were achieved during phase 1, 2, 3, and 4, respectively. It should be noted that effluent TSS concentration also decreased with the increasing COD removal rate. These results confirmed that leaching of dissolved organic compounds from the RRP was negligible or these compounds could be degraded by the biomass after about 30 days of acclimation period during start-up. Previous studies have shown the biodegradation of benzothiazole derivatives (De Wever and Verachtert., 1997; Gaja and Knapp.,1997; Haroune *et al*., 2002; Nawrocki *et al*., 2002). Contrary to high COD/BOD ratios found in the peat biofilter due to residual non-biodegradable organics, those of the RRP biofilter during phase 1 and 2 were most likely a result of fairly low concentrations of effluent BOD, since most of the biodegradable organic matter had been degraded and suspended solids were also removed by physical straining (Figure 5−7).



**Figure 5−8. Ammonia nitrogen concentration in the influent and effluent**


#### *Ammonia removal*

Organic nitrogen was converted into ammonia through the process of anaerobic decomposition in the septic tanks. Hence, effluent typically contains inorganic nitrogen primarily in the form of ammonium. Ammonia nitrogen concentrations  $(NH_3-N)$  in the influent ranged from 19 to 99 mg/L with an average value of 58.2 mg/L, and an average nitrate nitrogen concentration (NO<sub>3</sub>-N) was less than 15 mg/L (Fig. 5–8). Ammonia nitrogen would be adsorbed and oxidized to nitrate by autotrophic bacteria under aerobic conditions, which is referred to as nitrification. The RRP and gravel biofilter achieved excellent performance with respect to ammonia removal. The average ammonia nitrogen concentrations and removal efficiency from the RRP biofilter were 9 mg/L and 84%, respectively. Considering the nitrate nitrogen concentrations similar to those of influent, ammonia could be removed by the adsorption of ammonia on RRP or simultaneous nitrification and denitrification in the RRP biofilter. The nearly complete ammonia nitrogen removal was accomplished by nitrification in the gravel biofilter throughout the operation. This was reflected in the low ammonia nitrogen and increasing nitrate nitrogen concentrations (35 mg/L) of the gravel biofilter effluent. The nitrification process was probably enhanced by the sufficient void space of gravel media allowing for efficient diffusion of oxygen into biofilms. Ammonia nitrogen concentrations of the peat biofilter effluent increased and often exceeded those of the septic tank effluent during phase 2 and 3. The mean ammonia nitrogen removal efficiency dropped below 43% as nitrification was limited by oxygen availability due to the clogged filter during phase 3.



*Fecal coliform removal* 

The fecal coliform concentrations of the influent and effluent were determined by EC medium test kit from HACH. The levels of fecal coliform bacteria in the septic tank effluent ranged from 110 MPN/100 ml to 350/100 ml. These levels were much lower levels than those of typical septic tank effluent in the range of  $10^6$  to  $10^8$  MPN/100mL (EPA, 2002). The results showed that all three biofilters reduced the fecal coliform to less than 2 MPN/100mL. Therefore, RRP biofilter can be expected to perform similarly to other systems, such as peat filter or conventional gravel drainfield with respect to pathogen removal.

## **Conclusion**

Compared to a conventional gravel system and a peat biofilter system for treatment of septic tank effluent, the lab-scale RRP biofilter showed similar or better treatment performance in terms of organic removal and stable operation. After the start-up period, RRP biofilter achieved removal efficiencies for BOD5, TSS, ammonia nitrogen of 96%, 93%, and 90%, respectively, over the range of hydraulic loading rates of 1.4 to 5.0 gpd/ft<sup>2</sup>. On the other hand, ponded conditions often occurred in the peat biofilter which promoted anaerobic conditions and lower organic and ammonia removal. The operation was eventually discontinued at the end of phase 3 with a hydraulic loading rate of 4.0 gpd/ft<sup>2</sup> due to the persistent ponding problems. Suspended solids removal rates of the gravel filter did not depend on solids concentration in the influent, but hydraulic loading rates. High TSS concentrations in the effluent were assumed to be mostly biomass sloughed by hydraulic shear forces.



RRP provided high surface area and sufficient time for biological treatment. In addition, RRP provided a non-toxic media for biofilm attachment in biofilter. RRP was observed to provide ammonia adsorption capacity. Therefore, RRP biofilter is an acceptable leach filed media for treatment of septic tank effluent. Application of RRP as packing media of biofilter and also substitutes for natural aggregate in septic system drainfields would provide economic benefits.

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## **CHAPTER 6. CONCLUSIONS**

The performance and operational stability of the three pilot-scale SGBR for the treatment of industrial wastewater were investigated in this study. The two pilot-scale SGBR (R1 and R2) demonstrated excellent process performance for the treatment of slaughterhouse wastewater. R1 and R2 achieved the average COD removal rates of 94 and 95% at OLR ranging from 1.01 to 3.56 and 0.94 to 12.76 kg  $\text{COD/m}^3/\text{d}$ , respectively. During the operation of reactors, the solid retention times over 240 and 150 days for the R1 and R2, respectively were obtained. Long SRT enabled slow growing methanogens to get sufficient time to grow and stabilize, and promoted the proliferation of methanogenic bacteria in the granular sludge bed. The pilot-scale SGBR was also successfully employed for treating dairy processing wastewater under psychrophilic conditions. At low temperatures of 11°C, COD, BOD, and TSS removal rates obtained were 93, 96, and 90%, respectively. The SGBR achieved average COD, BOD, and TSS removal efficiencies higher than 91% even at high loading rates up to 7.31 kg  $\text{COD/m}^3/\text{d}$  with an HRT of 9 h. The of three pilot-scale SGBR were operating in a stable condition since pH values were in the optimal range and VFA/alkalinity ratios were fairly low throughout the experimental period. The average methane yield  $(0.26 \text{ L CH}_4/\text{g COD}_{\text{removed}})$  was possibly due to a high fraction of particulate COD (32 to 52%) and operation at low temperatures. Soluble COD seemed to be responsible for most of the methane production and particulate organic matter was physically retained by adsorption of the colloidal fraction of wastewater to granular sludge and the entrapment of coarse suspended solids in the sludge bed. Increased headloss through the granular bed due to the accumulated excess biomass and the retained solids were controlled by periodic backwashing.

A proper backwash rate is necessary to ensure effective removal of dispersed fine sludge



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and excessive suspended solids. Assuming that the average granule size and density in this study are in the range of 0.8-1.6 mm and 1000-1060 kg/m<sup>3</sup>, respectively, the minimum backwash rates varied from 0.02 to 4.34 m/h depending on the size and density of the granules. The proper backwash velocity ranged from 0.11 to 11.33 m/h based on the assumption that the bed porosity increased up to 0.4 and 50% expansion was selected as the optimum value. Therefore, backwash at a flow rate of 10-15 gpm (3.91-5.87 m/h) was applied to the pilot-scale SGBR (cross-sectional area: 6.25 ft<sup>2</sup>) treating dairy wastewater in Tulare, CA.

Compared to a conventional gravel system and a peat biofilter system for treatment of septic tank effluent, the lab-scale RRP biofilter provided similar or better performance in terms of organic removal and hydraulic capacity. After the start-up period, RRP biofilter achieved removal efficiencies for BOD5, TSS, ammonia nitrogen of 96, 93, and 90%, respectively, over the range of hydraulic loading rates of 1.4 to 5.0 gpd/ft<sup>2</sup>. On the other hand, the peat biofilter failed hydraulically and the gravel system showed high TSS concentrations in the effluent. RRP provided high surface area and sufficient time for biological treatment. In addition, RRP provided a non-toxic media for biofilm attachment in biofilter. RRP was observed to provide ammonia adsorption capacity. The results showed that RRP has the potential to be used as substitutes for natural aggregate such as gravel in septic system drainfields. The RRP biofilter can be used as alternative septic systems for the sites where an existing septic system has failed or site conditions, such as high groundwater table or small lot size, are not suitable for the installation of conventional septic systems.



#### **Engineering Significance**

The pilot scale SGBR was successfully employed for treating industrial wastewater under different operational conditions. The main advantages for the SGBR are high organic removal efficiency and operational simplicity. A high degree of organic removal was obtained in the SGBR even at short HRT and high OLR due to its long SRT. Consequently, capital costs are saved because of relatively small reactor volume sizes than other high rate anaerobic systems. The SGBR can also reduce relatively high costs associated with the packing materials, mixing equipment, or recirculation systems required. The SGBR generates methane which can be used in boilers or engine generators to produce electricity. Anaerobic treatment produces 11,000 BTU of methane per kg of chemical oxygen demand (COD) removed while aerobic treatment requires energy for aeration of 0.7 kilowatt-hour (kWh) per kg COD. Additionally, anaerobic processes generate only 20% of sludge compared with aerobic processes, resulting in significant cost saving for sludge handling, treatment, and disposal.

An aggregate such as gravel used in drainfield is not cost-effective due to the shipping cost for hauling gravel over long distances. According to industry averages, overall costs for 52 tons of gravel for one residential drainfield will be \$865 if it is assumed that the gravel is around \$10 per ton and the building site is 50-miles from the supplier. Peat filter media needs to be replaced since the peat decomposes and degrades over time. On the other hand, effective organic removal and stable operation of the RRP biofilter confirmed the feasibility of the septic tank effluent treatment. Application of RRP as substitutes for natural aggregate in septic system drainfields would provide substantial advantages in terms of cost saving due to their light weight. RRP is easy to handle without the use of heavy equipment, which reduces labor costs, limits



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damage to the property by machinery, and allows the systems to be constructed in locations

inaccessible to heavy equipment.

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#### **APPENDIX A**

#### *Ammonia adsorption ability*

Previous studies have shown that ground tire rubber was usually applicable to the removal of metals such as mercury and cadmium (Entezari *et al*., 2006; Manchón-Vizuete *et al*., 2005). Organic compounds sorption onto ground tires was also reported (Kim *et al*., 1997). Adsorption capacity of RRP for ammonia was investigated using batch adsorption tests. Various RRP dosages ranging from 0.2 to 5g were added into the Erlenmeyer flasks filled with 150mL of ammonium chloride solution with fixed concentration of  $10$ mg/L NH<sub>4</sub><sup>+</sup>. Amounts of ammonia adsorbed by RRP were calculated as the difference between amounts of ammonia initially added and those remaining in the supernatant solutions. The ability of RRP to adsorb ammonia was assessed using Langmuir and Freundlich isotherms.

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Ammonia concentration	Removed Ammonia $(mg/L as NH3-N)$			
	6.1			
4.7	5.1			
5.2	4.6			
5.9	3.8			
	2.8			
8.1	1.7			
9.8				
	$(mg/L as NH3-N)$ 3.7			

**Table A−1. Ammonia nitrogen removal in batch tests at different RRP dosage**

The most common adsorption isotherms are the Langmuir isotherm and the Freundlich isotherm. The linear form of the Langmuir isotherm is shown below:

$$
\frac{C_e}{Q_e} = \frac{C_e}{b} + \frac{1}{Kb} \tag{A.1}
$$



where  $Q_e$  = the amount of adsorbate adsorbed per unit mass of adsorbent (mg/g)

 $C_e$  = the concentration of adsorbate left in solution at equilibrium (mg/L)

 $K =$  the adsorption energy coefficient (L/mg)

 $b =$  the maximum adsorption capacity of adsorbent (mg/g)



**Figure A−1. Linear plot of Langmuir isotherm of ammonia adsorption on RRP**

The maximum adsorption capacity *b* and the adsorption energy coefficient *K* were determined by plotting *Ce*/*Q<sup>e</sup>* against *C<sup>e</sup>* as shown in Figure 5−9. However, a negative slope was obtained, which indicated that the adsorption behavior of RRP ammonia did not follow the assumption of the Langmuir isotherm possibly due to the heterogeneous surface of RRP. Therefore, the Freundlich isotherm was used since it was considered to be suitable for heterogeneous adsorption systems. The Freundlich adsorption isotherm can be expressed by following equation.



$$
Q_e = K_f \times C_e^{\frac{1}{n}} \tag{A.2}
$$

where  $Q_e$  is the amount of ammonia adsorbed per unit mass of RRP (mg/g) and  $C_e$  is the solution concentration at equilibrium, and  $K_f$  and  $n$  are the Freundlich constants relating to adsorption capacity and intensity respectively. A linear plot of log *Qe* against log *C<sup>e</sup>* is shown in Figure 5−9.

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$Q_e$ (mg/g)	$C_e$ (mg/L)	$\text{Log } Q_e$	$\text{Log } C_e$	
1.22	3.7	0.09	0.57	
1.7	4.7	0.23	0.67	
2.3	5.2	0.36	0.72	
3.8	5.9	0.58	0.77	
5.6	7.0	0.75	0.85	
8.5	8.1	0.93	0.91	

**Table A−2. Freundlich adsorption isotherm values**



**Figure A−2. Linear plot of Freundlich isotherm of ammonia adsorption on RRP**

The Freundlich constants  $(K_f \text{ and } n)$  were calculated using a linear regression method with correlation coefficients greater than 0.97 as shown in Figure 5–10. Constant *K*<sup>*f*</sup> was 3.65



mg/g and constant 1/n was 0.377. Therefore, the Freundlich isotherm was found to be suitable for describing adsorption behavior of RRP for ammonia nitrogen, and it could be expressed as the following:

$$
Q_e = 3.65 \times C_e^{0.377} \tag{A.3}
$$



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