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Evaluation of tire derived rubber particles as biofilter media and scale-up and design considerations for the Static Granular Bed Reactor (SGBR)

Jaeyoung Park
Iowa State University

Evaluation of Tire Derived Rubber Particles as Biofilter Media and Scale-up and Design

Considerations for the Static Granular Bed Reactor (SGBR)

by

Jaeyoung Park

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

Iowa State University

Ames, Iowa

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EXECUTIVE SUMMARY

Research demonstrated three different bioreactors to evaluate use of tire rubber as biofilm attachment media in bioreactors for wastewater treatment: aerobic biofilter, anoxic bioreactor, and a hybrid anaerobic static granular bed reactor (SGBR). In addition, owing to the results from non-toxicity to microorganism and good surface area for biofilm attachment, size distribution, chemical composition, scanning electron microscopy, and whole effluent toxicity analyses verify the potential of TDRP (tire derived rubber particles) usage for biofilm attachment media. The trickling filter system using chunk rubber (average diameter of approximately 3 cm) achieved 79.6-90.1% COD removal efficiency at organic loading rates ranging from 0.12 kg COD/m^3·d to 0.34 kg COD/m^3·d. The hybrid SGBR and anoxic TDRP filter filled with fine rubber particles (average particle diameter of approximately 0.2 mm) achieved 90-97% of COD removal and above 97% of nitrogen removal, respectively at various hydraulic retention times of 48 to 20 h. The utility of TDRP media in multiple biofiltration applications was demonstrated by the performances of three TDRP biofilm media systems and analysis of TDRP characteristics.

The biofilter system filled with TDRP filter media was utilized to treat the odorous gas contaminant, hydrogen sulfide. This bioreactor system achieved over 94% removal efficiency at 20-90 ppm of inflow H_2S concentration while operating in 20-67 seconds of EBRTs, indicating that overall effective operation was performed at mass loading rates of

H_2S ranging from 19.6 to 28.5 g H_2S/ m^3 /hour. It was apparent by the effectiveness of the system's performance that this system had the capability to hydrogen sulfide.

Performance between the hybrid SGBR with the addition of TDRP and SGBR reactors was compared to validate the ability of TDRP media as a substitute for granules. Both systems showed similar high COD removal efficiencies (over 95%) at hydraulic retention times of 48 to 12 hours and resulting organic loading rates of 1 kg/m^3/d to 4 kg/m^3/d. The applicability of TDRP media to the bioreactor was also shown by the differences in performance between reactors with and without TDRP addition in the same granular sludge volume.

An on-site pilot-scale SGBR system was evaluated for treating slaughterhouse wastewater from a food plant in Iowa to provide treatability and compared to other high-rate anaerobic systems and critical elements for commercialization. High organic removal efficiency (over 95% of TSS and VSS removal) was obtained due to the consistent treatability of SGBR system during operation at HRTs of 48, 36, 30, 24, and 20 hours. An effective backwash procedure was performed to waste a portion of the accumulated solids in the system. This procedure limited the increase in hydraulic head loss and maintained the system stability. COD removal efficiencies greater than 95% were achieved at organic loading rates ranging from 0.77 kg/m^3/d to 12.76 kg/m^3/d. This performance was consistently better than other high-rate anaerobic systems treating slaughterhouse wastewater.

CHAPTER 1. GENERAL INTRODUCTION

Tire Reuse

Billions of tires are discarded annually, creating increased interest in the development of rubber reuse alternatives. Currently, there are an estimated 2-3 billion scrap tires stockpiled in the U.S. Some of the current reuse alternatives for used tires include tire derived fuel (TDF), highway embankments, asphalt, and molded products (Azizian et al., 2003; Navaro et al., 2004; Sunthonpagasit and Duffey, 2004). Environmental applications for tire rubber have mainly been in adsorption systems. Manchón-Vizuete et al. (2005), for instance, tested chemically- and heat-treated tire rubber for its ability to adsorb mercury. Entezari et al. (2006) used ground tire rubber, preconditioned with ultrasonic vibrations, to remove cadmium from aqueous solutions. Other experimental applications for crumb rubber include its use as a ballast water filtration media (Xie and Chen, 2004; Tang et al., 2006), subsurface drainage for nutrient mitigation (Lisi et al., 2004), and septic tank liners.

Hydrogen Sulfide Control

Waste gases from a number of industrial plants, waste disposals, composing plants, and wastewater treatment plants emit unwanted odors containing sulfur compounds, namely hydrogen sulfide (H_2S) and mercaptans as the principal source (Burgess et al., 2001). Odorous gases should be removed for reasons of public health, safety, and the prevention of corrosion (Jensen and Webb, 1995). Over 10 ppm of hydrogen sulfide can affect the human

health and fatal problems can be raised at higher than 600 ppm (Droste, 1997). Hence,

various odor removal systems are used at sites producing odorous gases.

Recently, biological treatment has been investigated and developed for H_2S control,

since commonly used chemical scrubbers have several significant disadvantages such as high

maintenance costs and hazardous chemical usage (Gabriel et al., 2004; Hansen and Rindel,

2000). Several biological methods of odor treatment such as bioscrubbers, biotrickling

filters, and biofilters have been developed and used with good results (Morton et al., 2005;

Nishimura and Yoda, 1997; Wolstenholme et al., 2005).

Static Granular Bed Reactor (SGBR)

In addition to developing markets for tire rubber from scrap tires, this research

focuses on the scale-up and design considerations of a new anaerobic treatment technology.

These include static granular bed reactor (SGBR) for its potential in renewable energy

production.

Concept

The SGBR is a simple downflow anaerobic system developed at Iowa State

University. It utilizes a bed of active anaerobic granules in a downward flow regime (Figure

1-1). The innovation for this reactor configuration is that it uses highly active anaerobic

granules (just as in a UASB system), but it operates in a downflow mode, eliminating the

need for the GLSS (gas, liquid, solids separator) required in UASB systems. Other reactor

configurations use a downward flow regime (e.g., the anaerobic filter), but the SGBR is the

first granular sludge system to operate in a downflow mode. This configuration allows for

exceptional effluent quality, simple operation, and reduced volume requirements.
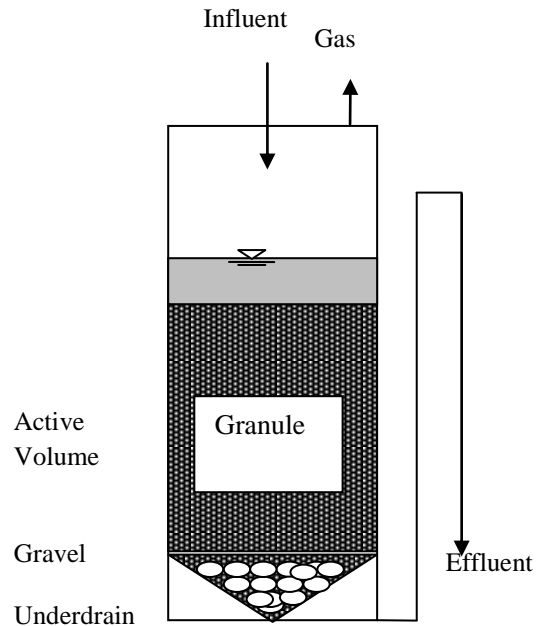
Figure 1-1. Schematic of SGBR

The advantage of a downflow configuration is the generated biogas rises and is easily

separated from the granules and the liquid at the top of the reactor. Granule buoyancy is not

a detriment to process performance in the SGBR, as in the UASB. In contrast to the UASB,

there is no need for a sophisticated three-phase solids, gas, and liquid separator or

recirculation pumps, timers, mixers, and any other ancillary equipment required for UASB

systems. Consequently, the effluent quality of the SGBR is improved in comparison to the

UASB. The biomass granules are retained within the reactor by the use of a gravel

underdrain. As a result, temperature and hydraulic loading changes are not expected to

significantly affect effluent quality.

Feasibility

The performance of the SGBR has been demonstrated in numerous laboratory and pilot studies. The SGBR has been used to treat synthetic wastewater consisting of non-fat dry milk, municipal wastewater, pork slaughterhouse wastewater, and landfill leachate with excellent results (Mach and Ellis, 2000; Roth and Ellis, 2004; Evans and Ellis, 2005; Debik et al., 2005). Moreover, the SGBR was also tested at low temperatures, e.g., 8 and 15 ºC, for municipal wastewater treatment (Evans, 2004).

Study Objective

The focus of this research is to evaluate the biofilter system with TDRP media as a potential reuse alternative for scrap tire rubber, find the appropriate applications of the TDRP biofilter system, and evaluate the treatability of this filter system compared to the current media. An additional objective investigates scale-up and design parameters for the SGBR treating slaughterhouse wastewater. Several various biofilter systems using TDRP media have been applied to wastewaster treatment at various organic loading rates. To achieve optimum removal of hydrogen sulfide, the biofilter with TDRP was utilized. The evaluation of a pilot-scale SGBR was performed to provide some critical elements and highlight the commercialization potential of treating slaughterhouse wastewater.

Dissertation Organization

This dissertation is organized into four major parts with individual papers. The first part evaluates use of tire rubber as biofilm support media in bioreactors for wastewater

treatment: aerobic biofilter, anoxic bioreactor, and a hybrid anaerobic static granular bed

reactor (SGBR). The second part demonstrates odorous gas (e.g. hydrogen sulfide) treatment

with the TDRP biofilter. The next part is a comparison study between SGBR and hybrid

SGBR to determine the suitability of TDRP as biofilm support media. The final part

establishes the feasibility of treating slaughterhouse wastewater with the SGBR via a pilot

scale system.

CHAPTER 2. EVALUATION OF TIRE DERIVED RUBBER

PARTICLES FOR BIOFILTRATION MEDIA

Jaeyoung Park and Timothy G. Ellis
Department of Civil, Construction, and Environmental Engineering,
Iowa State University, Ames, Iowa, 50011 U.S.A.
A paper corresponding to a presentation at WEFTEC 2006

Introduction

Currently, on the average, one tire is discarded every year in the U.S. for every living man, woman, and child. While the reuse market for used tires has increased over the years to approximately 75%, there are still an estimated 2-3 billion used tires stockpiled in the U.S. The largest demand (33% of the tire reuse market) for used tires is in tire derived fuel (TDF), primarily for use in cement kilns (Sunthonpagasit and Duffey, 2004). Other current markets for used tires include civil engineering (CE) applications (15%) and crumb rubber (12%). CE applications for used tires include leachate collection and recovery systems (see Phaneuf and Glander, 2003) and highway embankments. Approximately one third of crumb rubber produced is used for asphalt modification (e.g., crumb rubber asphalt concrete, see Azizian et al., 2003 and crumb tire rubber bitumens, see Navarro et al., 2004). Another third of the crumb rubber is used for molded products (e.g., using crumb rubber in lieu of virgin rubber). Additional uses for crumb rubber include sports and horse arena surfaces, automotive products, and landscaping mulch (Sunthonpagasit and Duffey, 2004).

Environmental applications for tire rubber have mainly been in adsorption systems. Manchón-Vizuete et al. (2005), for instance, tested chemically- and heat-treated tire rubber

for its ability to adsorb mercury. Entezari et al. (2006) used ground tire rubber, preconditioned with ultrasonic vibrations, to remove cadmium from aqueous solutions. A review by Mui et al. (2004) suggested that activated carbon material made from waste tire rubber could result in porosities in excess of 40% of pore volume and surface areas over 1000 m^2/g. Other experimental applications for crumb rubber include its use as a ballast water filtration media (Xie and Chen, 2004, Tang et al., 2006), subsurface drainage for nutrient mitigation (Lisi et al., 2004), and septic tank liners.

The objective of this study was to evaluate TDRP (tire derived rubber particles) from Envirotech Systems, Inc. as a suitable media for biological growth and biofilm development in anaerobic, aerobic, and anoxic environments. In this study, three different types of reactors were constructed and operated—a trickling filter with effluent recycle, a denitrification filter with fixed media for attached growth, and a hybrid-static granular bed reactor with anaerobic granular sludge and TDRP. Each system was typical of what might be used in the field with the exception of the hybrid-SGBR.
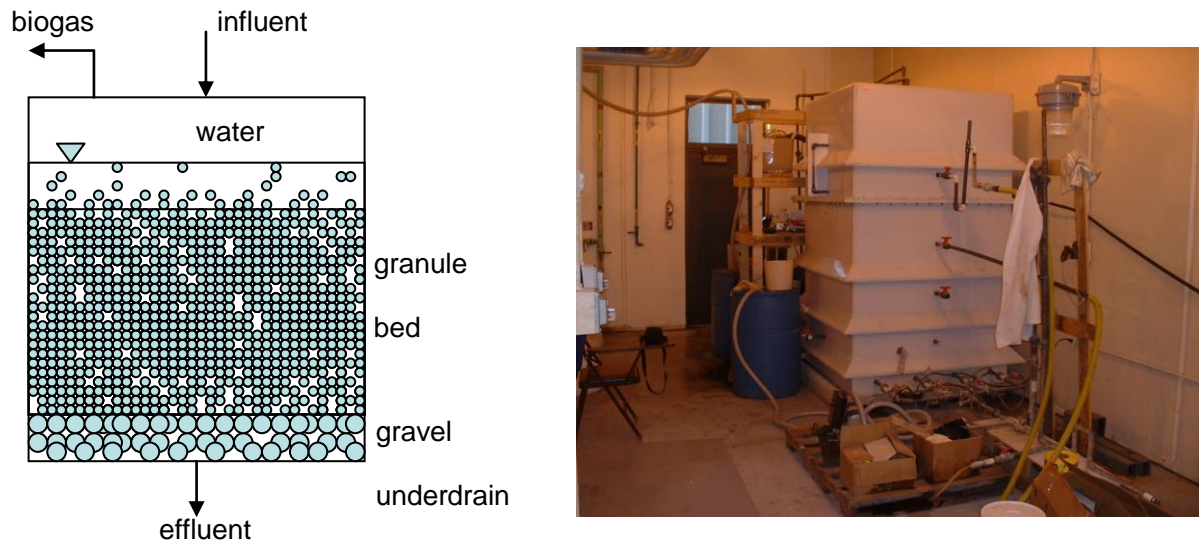
Figure 2-1. Schematic and photo of a pilot-scale SGBR (U.S. patent #6,709,591).

The SGBR is a simple downflow anaerobic system developed at Iowa State University. It utilizes a bed of active anaerobic granules in a downward flow regime (see Figure 2-1). The innovation in this reactor configuration is that it uses highly active anaerobic granules (just as in a UASB system), but it operates in a downflow mode. The advantage of a downflow configuration is the generated biogas rises and is easily separated from the granules and the liquid at the top of the reactor. Granule buoyancy is not a detriment to process performance in the SGBR as in the UASB. In contrast to the UASB, there is no need for a sophisticated three-phase solids, gas, and liquid separator. Neither is there a need for recirculation pumps, timers, mixers, nor other ancillary equipment required for the UASB systems. Consequently, the effluent quality of the SGBR is improved in comparison to the UASB.   The biomass granules are retained within the reactor by the use of

a gravel underdrain. Consequently, temperature and hydraulic loading changes are not expected to significantly affect effluent quality.

The technological innovation of the SGBR is that it uses highly active anaerobic granular biomass in a downflow configuration. Other reactor configurations use a downward flow regime (e.g., the anaerobic filter), but the SGBR is the first granular sludge system to operate in a downflow mode. This configuration allows for exceptional effluent quality, simple operation, and reduced volume requirements. The performance of the system was demonstrated in numerous laboratory and pilot studies on a variety of wastewaters (Mach and Ellis, 2000, 2001, Roth and Ellis, 2004, Evans and Ellis, 2005, Debik et al., 2005). The addition of TDRP to the granule bed was evaluated in this study to determine its suitability as a media and to offset the high cost of anaerobic granules (which traditionally have sold for approximately $66/m^3).

Materials and Methods

The TDRP material was obtained from Envirotech, Inc. in three samples. Two of the samples were fine rubber and one of the samples was large chunk rubber. Size distribution analysis was carried out using sieve analysis and light microscopy. A wet sieve was used to characterize the fine shredded tire rubber. The sieve test was preformed using a wet sieve machine with about 100 g samples. Nine sieves were arranged from the largest opening to the smallest, and allowed all possible materials to pass through each sieve to distribute by particle size. The large chunk rubber was measured directly with a ruler to determine an

average approximate size. Physical and chemical analysis were conducted, using a scanning

electron microscope (Hitachi, S-2460N). The samples were applied to a carbon based disc

and inserted under the electron stream. The elements were identified in spots or over a larger

area of the sample based refracted waves from the material.

Toxicity Test

Toxicity testing was performed by the University of Iowa's Hygienic Laboratory,

Iowa's only laboratory certified to conduct the whole effluent toxicity (WET) test. Lethal

concentrations (LC_{50}) were determined for WET organisms—*Pimephales promelas* and

Ceriodaphnia dubia. Samples for testing were obtained by adding 100-g of the TDRP

material to 4-L mixed liquor samples obtained from the Boone (Iowa) Water Pollution

Control Plant. Two separate samples of TDRP material were tested, Product A and Product

B (Product B had small metal flakes mixed in with the rubber particles). After dosing the

mixed liquor with TDRP, the samples were stirred with a standard jar test apparatus at 120

rpm for 30 min. Supernatant from the mixed liquor/TDRP samples and the control sample

(mixed liquor without TDRP) were sent to the State Hygienic Laboratory for analysis.

Biofiltration systems setup

Three laboratory scale biofilter reactors were designed and operated to evaluate the

TDRP for the biofiltration media in various operating conditions as shown in Figure 2-2.
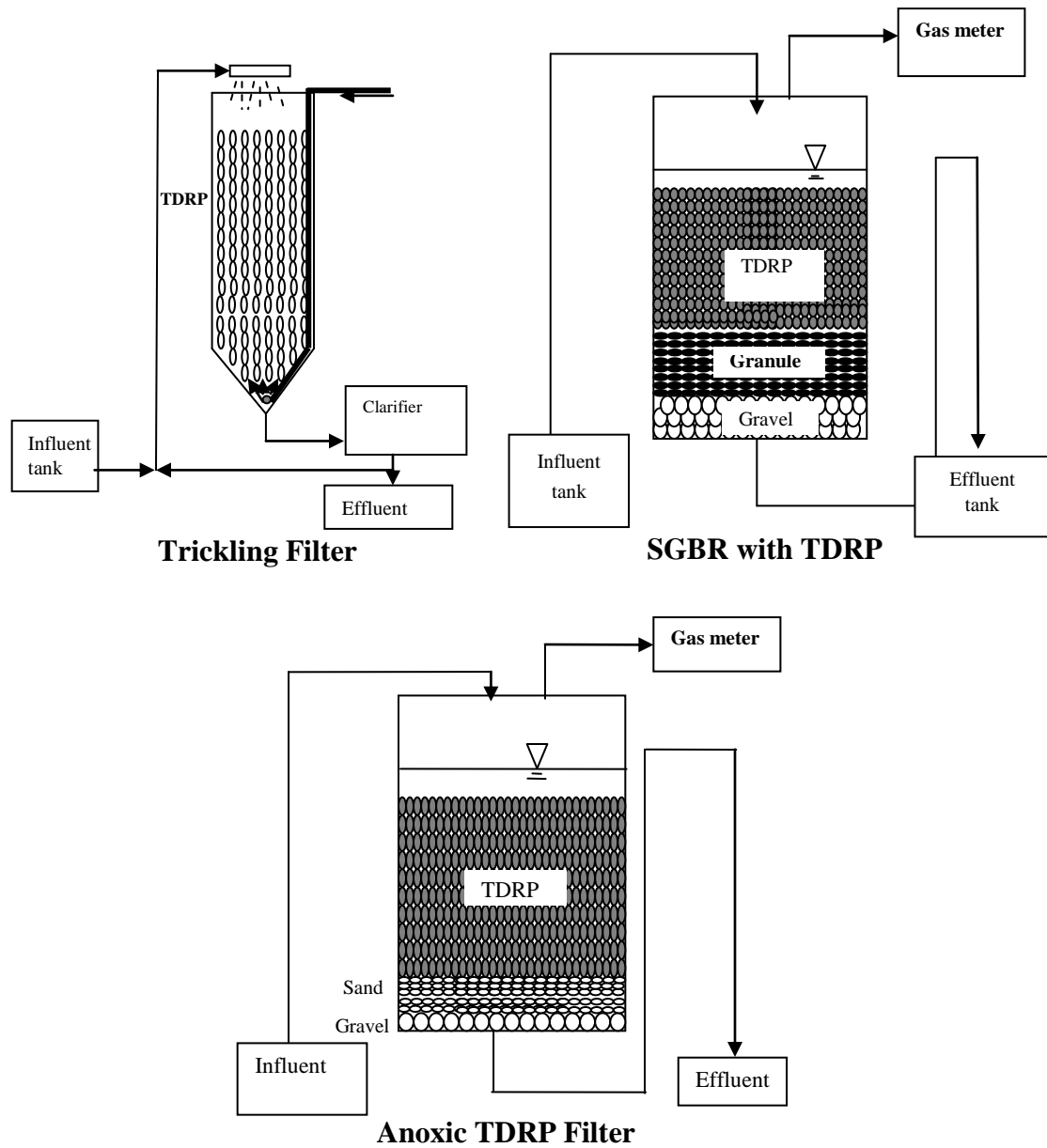
Figure 2-2. Configuration of three biofiltration systems applied with TDRP

The laboratory-scale trickling filter was fabricated from Plexiglass with a working

volume of 10-L filled with coarse (chunk rubber) TDRP. Forced air (upflow) was utilized to

ensure aerobic conditions. The trickling filter was seeded with biomass from the Boone

Water Pollution Control Plant, and 100% recirculation of effluent was employed. Synthetic

wastewater was fed to the trickling filter at a concentration of 300 mg COD/L. For the

hybrid-SGBR the active volume was 1.5-L and gravel was used for the underdrain at the base

of the reactor. The hybrid SGBR was seeded with 0.5-L anaerobic granules and 1.0-L fine

TDRP. During operation, various hydraulic retention times were applied to the hybrid SGBR

fed with non-fat dry milk as the influent substrate. Finally, a 1.5-L active volume downflow

denitrification filter was constructed and evaluated. Gravel was used at the base of this

reactor followed by 0.4-L fine sand and 1-L fine TDRP. This reactor was fed synthetic

wastewater including non-fat dry milk as carbon source and potassium nitrate (KNO_3) as the

nitrate source. Feed composition for each of the three bioreactors is shown in Table 2-1.

Table 2-1. Composition of synthetic influent solution

	Trickling Filter	SGBR with TDRP	Anoxic TDRP filter
NFDM (g/L)	0.3-0.6	1.1-1.9	0.1-0.4
$NaHCO_3$ (g/L)	0.1-0.2	0.3-0.7	0.03-0.1
$FeCl_3$(mg/kg of NFDM)	4.6	4.6	4.6
$ZnCl_2$(mg/kg of NFDM)	1.0	1.0	1.0
$NiCl_2 6H_2O$ (mg/kg of NFDM)	0.8	0.8	0.8
$CoCl_2 6H_2O$ (mg/kg of NFDM)	3.0	3.0	3.0
$MnCl_2 6H_2O$ (mg/kg of NFDM)	15.0	15.0	15.0
KNO_3 (g/L)	-	-	0.4-0.7

Results and Discussion

TDRP Characteristics

The size distribution and chemical tests are very important to the potential uses of a material. Knowing the sizes and distribution of particles helps determine reasons for or against certain characteristics of the TDRP. A chemical analysis can further help to predict characteristics and problems that may occur. The fine TDRP used for these tests had fairly similar fraction in each size distribution as evidenced by the consistent slope of the line representing the size of particles as shown in Figure 2-3. This could help predict the use of this material as the filter media. The chunk rubber had a mean size of 2.6±0.8 cm in length and 0.8±0.4 cm in width.
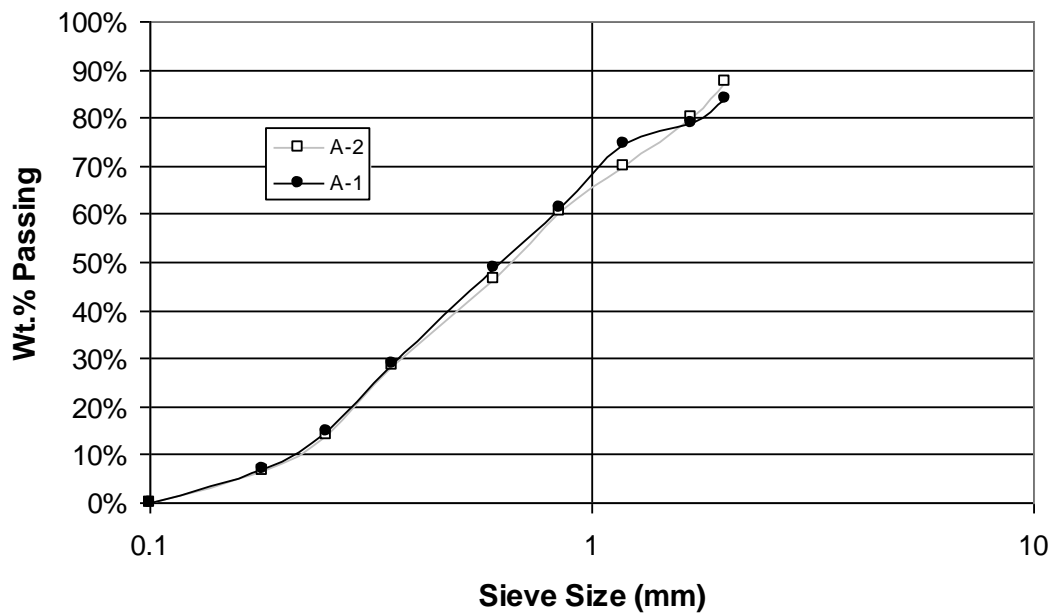
Figure 2-3. Size distribution of the fine TDRP sample, Product A (fine TDRP)

Using SEM, it was possible to detect elements present in the TDRP. Chlorine was present in both small and large areas tested. Sulfur was also found, which was expected, since sulfur is used to manufacture tires. In some of the areas tested, sulfur spiked to many times that of the other chemicals. Silicon, calcium, and oxygen were also observed, but at lower amounts than many of the other elements. Zinc and magnesium were also detected in significant amounts.

Table 2-2 shows the results of the WET for two TDRP samples tested. The two products did not show significant toxicity. It was not possible to calculate the median lethal concentration (LC_{50}) value, due to insufficient mortality of the *Pimephales promelas* and *Ceriodaphnia dubia*. This analysis found no toxicity in the *Pimephales promelas* and *Ceriodaphnia dubia* for product "A," fine TDRP, which did not have any dead of either species. However, the product "B," fine TDRP with metal addition, had some dead species during the test, and the *Pimephales promelas* had a higher mortality than *Ceriodaphnia dubia* for product "B." Therefore, product "B" seems to have a higher mortality than product "A," owing to the addition of metal. However, product "B" also did not show sufficient mortality to conclude an LC_{50} concentration for this material.

Table 2-2. Mortality data at 48 hours

Concentration	Product"A" (number dead/ number tested)		Product "B" (number dead/ number tested)	
	Pimephales promelas Mortality	*Ceriodaphnia dubia* Mortality	*Pimephales promelas* Mortality	*Ceriodaphnia dubia* Mortality
Lab control	0/20	0/20	0/20	0/20
Control	3/20	0/20	3/20	0/20
6.25%	0/20	0/20	2/20	2/20
12.5%	0/20	0/20	3/20	0/20
25 %	0/20	0/20	7/20	0/20
50 %	0/20	0/20	3/20	2/20
100 %	0/20	0/20	4/20	4/20

TDRP Trickling filter

To determine the TDRP trickling filter reactor performance, the trickling filter was operated at different organic loading rates ranging from 0.12 to 0.34 kg COD/m^3·d. During operation, 100% recirculation was utilized to increase removal efficiencies and dissolved oxygen concentrations in the trickling filter. Recirculation also reduced the clogging potential by increasing the hydraulic loading and dilution of the influent COD concentration. Figure 2-4 shows influent and effluent COD concentrations and the removal efficiency of the trickling filter reactor. Once the biofilm became established on the TDRP media, the COD removal efficiency in the trickling filter remained high. Increasing hydraulic and organic loading had little effect on the performance of the system.
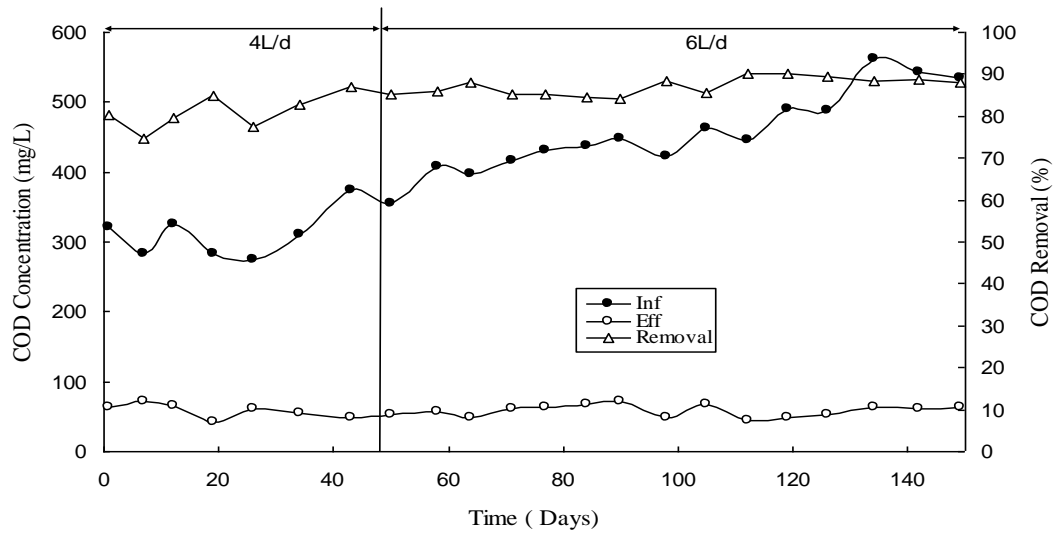
Figure 2-4. COD variation and removal efficiency in the TDRP trickling filter.

During operation, the pH of the trickling filter remained between 6.7-7.3 in the

influent and 6.9-7.4 in the effluent (Table 2-3). This study achieved low suspended solids

concentrations in the effluent from the trickling filter, ranging from 5 to 10 mg TSS/L and 4

to 8 mgVSS/L owing to the clarifier. The low SS values can be explained by the good

settlability of the sloughed biomass from the TDRP medium in the reactor. Moreover, the

BOD removal efficiency of this system was greater than 90%, indicating good capability of

TDRP as biofilm support material.

Table 2-3. Summary of the results from the trickling filter reactor

Hydraulic loading rate	4 L/d*		6 L/d*	
	Influent	Effluent	Influent	Effluent
COD(mg/L)	310 ± 34.3	58 ± 10	456 ± 57.8	58 ± 8.5
BOD$_5$(mg/L)	-	-	381 ± 6	19.8 ± 2
pH	7.06 ± 0.18	7.20 ± 0.14	7.2 ± 0.07	7.37 ± 0.08
TSS(mg/L)	106 ± 21.7	7.5 ± 3.1	132 ± 22.8	7.9 ± 4.8
VSS(mg/L)	89 ± 30.4	5.3 ± 2.9	104 ± 32.4	6.1 ± 3.4
Alkalinity (mg/L as CaCO$_3$)	311 ± 56.4	320 ± 42	315 ± 41.8	322 ± 43

* The flowrates of 4 and 6 L/d corresponds to hydraulic loading rates of 26 L/m^2

Hybrid SGBR with TDRP

The hybrid SGBR augmented with TDRP maintained a good treatment performance during the operating period. Figure 2-5 shows the COD values of the influent and effluent and its removal efficiency during the operating period in this study. The SGBR augmented with TDRP consistently removed 90-97% COD at HRTs of 48 to 20 hour while the influent COD concentration and organic loading rate increased from 0.4 to 3 kg/m^3·d. Effluent COD gradually decreased and remained below 100 mg/L after the initial start-up. Moreover, effluent BOD was below 20 mg/L and BOD$_5$ removal efficiency was over 96% at the 20-h HTR condition as shown in Table 2-4.
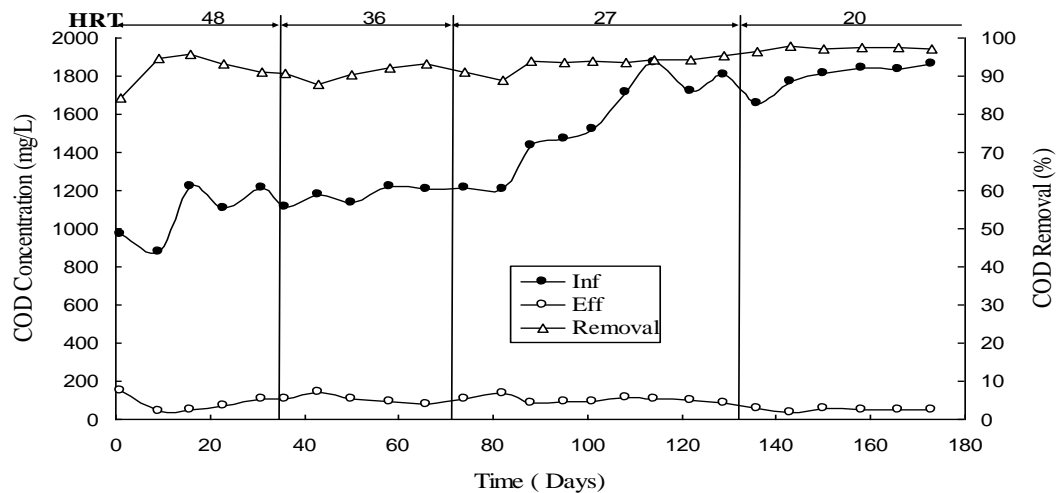
Figure 2-5. COD variation and removal efficiency in hybrid SGBR (anaerobic)

Effluent pH, VFAs, and alkalinity averaged 7.46, 11.24 mg/L, and 649 mg/L as CaCO$_3$, respectively (Table 2-4). Effluent VFA concentrations remained below 15 mg/L at all HRT conditions (except during the initial start-up period), indicating the high degree of stability of this system. The consistent performance of the SGBR augmented with TDRP is

further evidence of the ability of the TDRP material to support biofilm attachment under

anaerobic conditions.

Table 2-4. Summary of average values of the results from the SGBR reactor

HRT	48 hour		36 hour		27 hour		20 hour	
	Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent
COD(mg/L)	1078±150	86±44	1161±46.5	113±21	1518±155	99±16	1798±75.4	50±7.5
BOD$_5$(mg/L)	-	-	-	-	-	-	1230±15	18.2±2.1
pH	7.51±0.08	7.56±0.10	7.30±0.05	7.39±0.11	7.27±0.05	7.44±0.10	7.32±0.19	7.46±0.16
TSS(mg/L)	140±16.7	45±11.2	168±15.8	35±8.2	186±18.8	25.2±10.8	216±28.4	28.6±6.8
VSS(mg/L)	130±22.3	35±8.9	143±32.1	25±6.9	174±21.1	23.5±5.9	194±31.6	24.3±11.2
Alkalinity (mg/L as CaCO$_3$)	286±36	546±129	270±20	665±41	282±31	665±55	272±23	7006±28
VFAs(mg/L)	59.4±6.3	13 ±8.9	63.9±15.3	11 ±1.1	83.7±5.4	12 ±2.3	99.1±6.3	8 ±0.8

Cumulative methane production is given in Figure 2-6. Actual cumulative methane

production was calculated using the measured methane content of the biogas generated by

the SGBR with the TDRP reactor. The average methane content of produced biogas was

73.1% during this study. The theoretical methane production was calculated from the daily

removed COD, assuming complete conversion of COD removed from the influent (0.35 L /g

removed COD). At long HRTs, such as 48 hours, the actual cumulative methane was close

to the theoretical production, since there was little methane lost from the effluent. The

disparity between actual and theoretical cumulative methane appeared at HRTs shorter than

36 h. This was likely due to the solids accumulation in the system. However, the trend of

the two values was similar, and the cumulative methane production corresponded adequately

to the theoretical value with excellent methane content in the biogas.
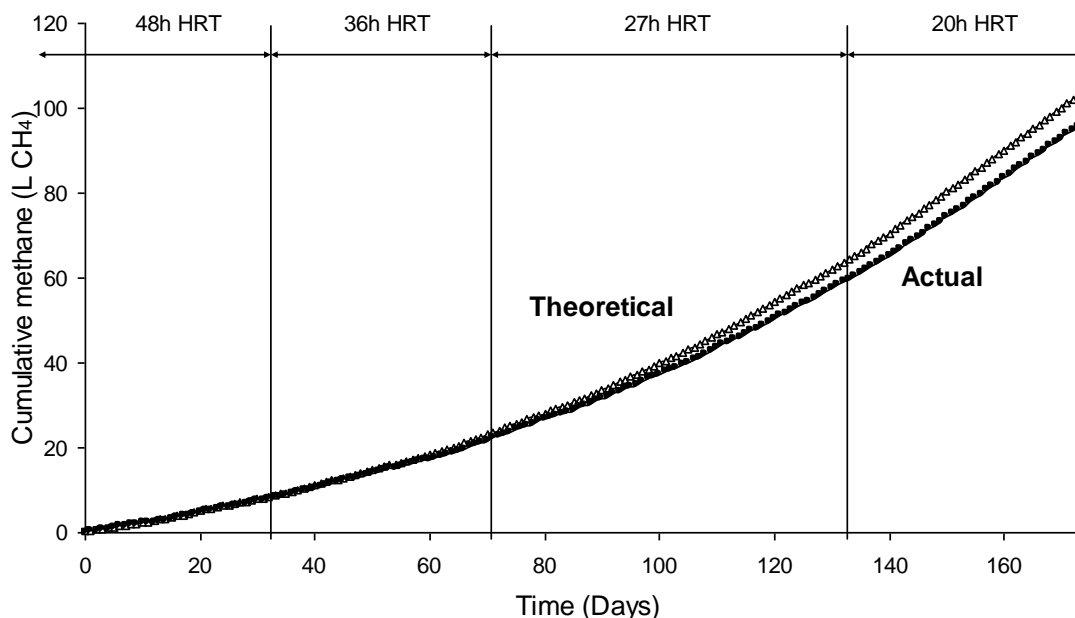
Figure 2-6. Cumulative actual and theoretical methane production in SGBR

<u>Anoxic TDRP filter</u>

TDRP was used in the denitrification filter to evaluate its suitability in anoxic environments. In the anoxic TDRP reactor, COD removal efficiency was less than 80%, mainly due to the fact that COD was supplied in excess of the nitrate added (Figure 2-7) to achieve full denitrification. Consequently, the nitrate-nitrogen concentration in the effluent was low, ranging from 0.8-2.1 mg/L (Table 2-5). In addition, the BOD_5 concentration in effluent was below 10 mg/L, indicating most of the easily biodegradable COD was removed in this reactor. Effluent TSS and VSS concentrations were below 15 mg/L at various HRTs conditions. Alkalinity increased in the effluent, owing to its recovery by denitrification.

Table 2-5. Summary of average values of the results from the Anoxic reactor

HRT	48 hour		36 hour		27 hour	
	Influent	Effluent	Influent	Effluent	Influent	Effluent
COD(mg/L)	398+292.9	101+138.3	184.6+10.0	40.09+7.54	140.3+39.5	39.1+5.8
BOD$_5$(mg/L)	-	-	-	-	42+4.8	8.9+2.4
pH	7.29+0.03	7.78+0.14	7.23+0.15	7.75+0.15	7.26+0.1	7.84+0.09
TSS(mg/L)	732+1.3	17.5+9.1	52.0+ 9.2	12.3+ 3.1	46.2+5.8	7.5+ 2.3
VSS(mg/L)	601+9.8	15.1+8.3	44.3+ 4.9	10.2 +2.8	39.1+ 4.6	5.0 +0.9
Alkalinity (mg/L as CaCO$_3$)	276.7+27.8	586.7+243.7	254.4+26.5	803.3+91.1	257.5+17.1	860.0+51.6
Nitrate(mg/L)	73.9+16.6	1.5+0.5	87.1+6.28	1.8+0.3	92+1.3	2.0+0.2
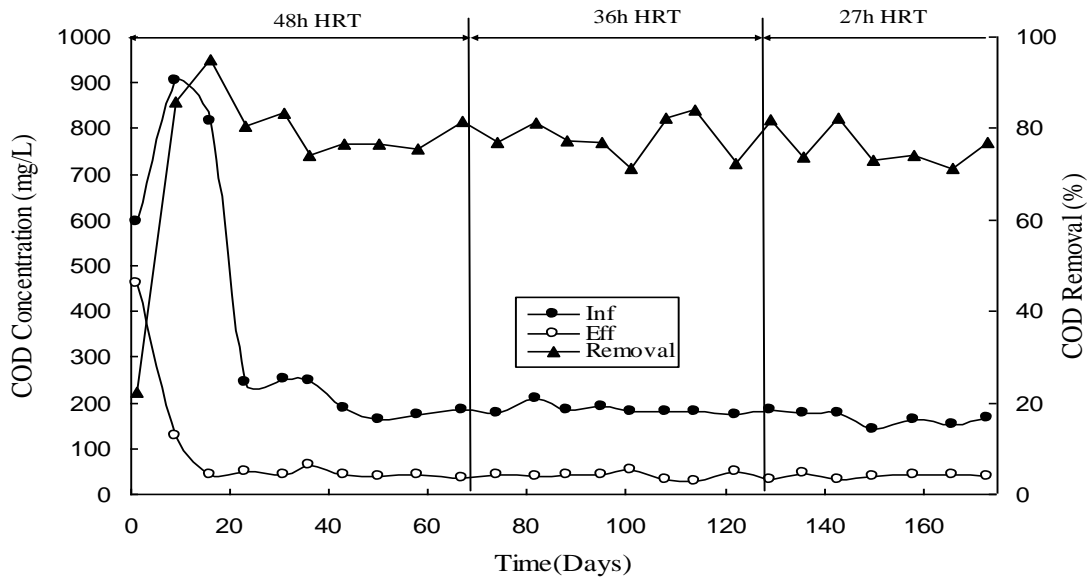
Figure 2-7. COD variation and removal efficiency in Anoxic TDRP filter

Figure 2-8 shows the nitrate-nitrogen concentration of the influent and effluent and its removal efficiency in this study. The nitrate-nitrogen concentration was increased from 52 mg/L to 94 mg/L at various HRTs to evaluate the denitification ability of this TDRP filter system. The C/N ratio also decreased from 4.7 to 1.6 as the nitrate increased. Nitrogen removal efficiency was above 97% during the operating time which indicates the high potential of TDRP as biofilter support media for denitrifying bacteria.
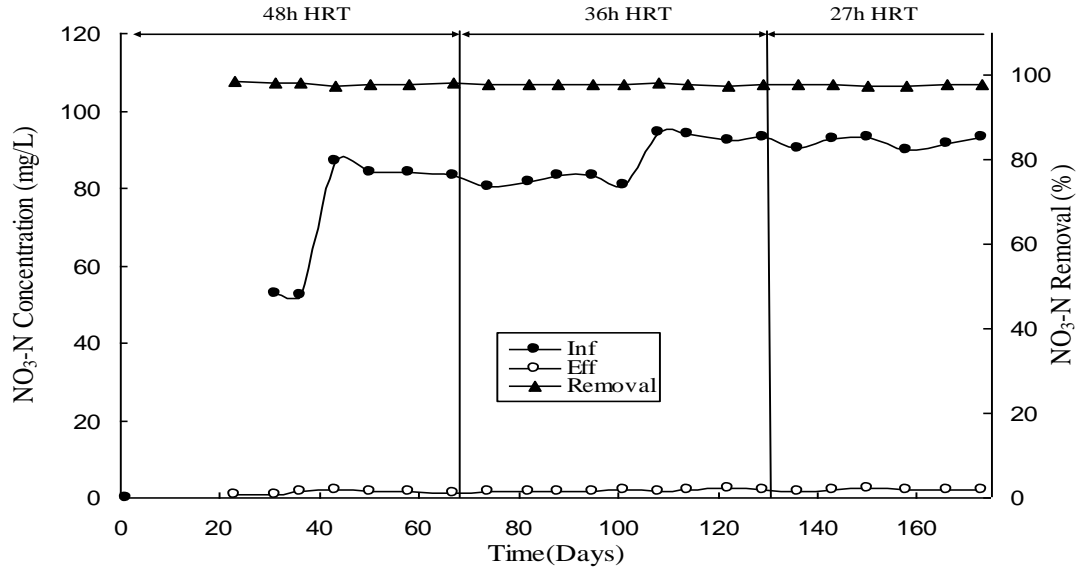
Figure 2-8. Nitrate variation and removal efficiency in Anoxic TDRP filter

SEM (scanning electron microscopy) Analysis

Samples of the TDRP media were randomly collected from the trickling filter and tested with scanning electron microscopy (SEM) to obtain high resolution images of the biofilm. The collected TDRP media had abundant microbial growth on each surface as shown in Figure 2-9. The most distinctive bacteria on the medium appeared to be similar to *Chroococus,* which are cyanobacteria-like organisms that form aggregates of two to four cells. There were also other cyanobacteria such as *Gloeocaps*a on the medium. Some fibrous spots were found on the surface of TDRP, which also supplied a growth area for the microorganisms.
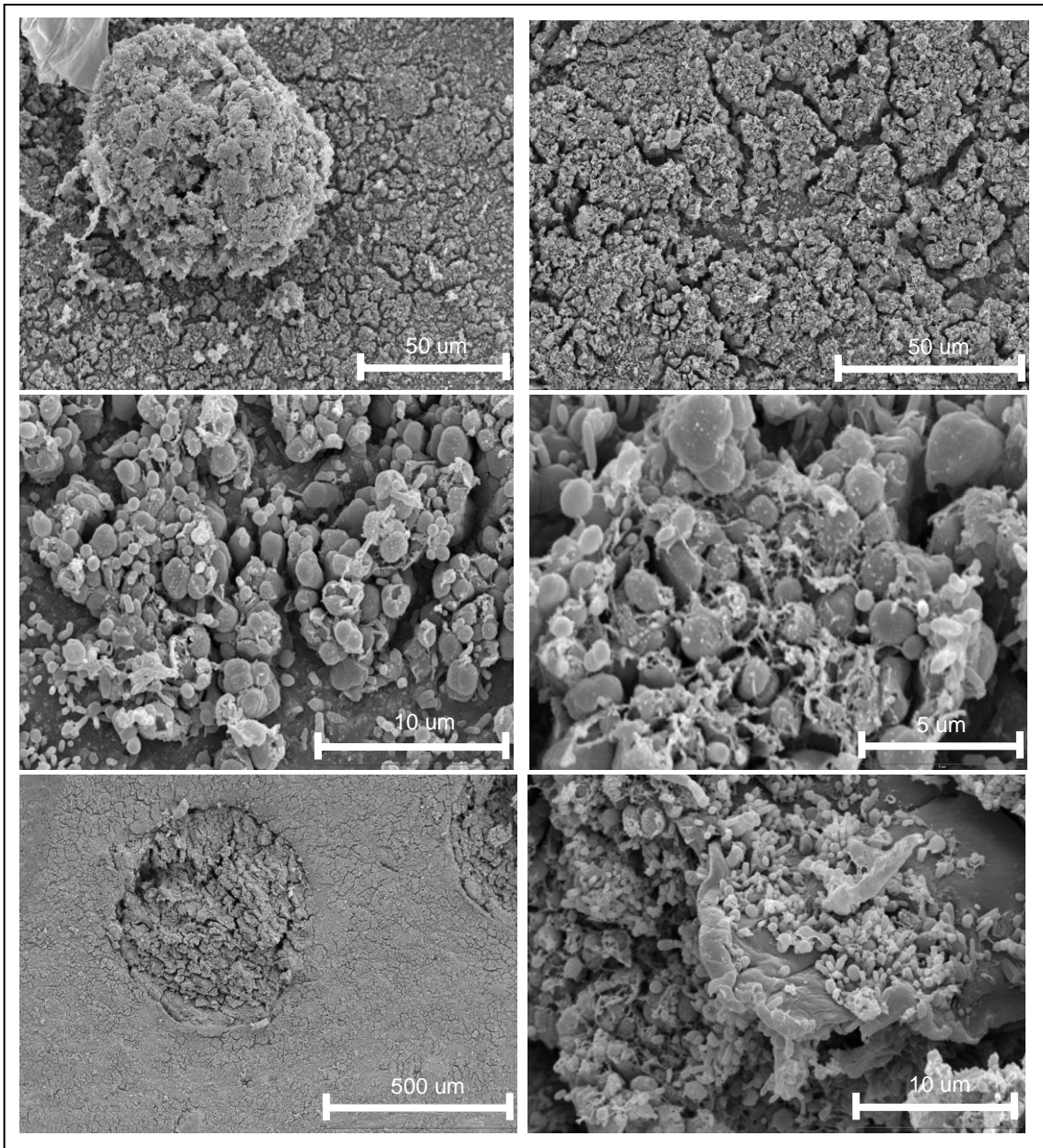
Figure 2-9. SEM picture of TDRP filter medium

Conclusions

Results from each aspect of this study showed the high potential of TDRP to support biological activity in a variety of wastewater treatment applications. From the toxicity test it was observed that the supernatant from mixed liquor in contact with TDRP did not demonstrate a median lethal concentration (LC_{50}) since there was no substantial mortality of tested species. The size distribution analysis and chemical tests provided a good basis for the use of TDRP as biofilter media.

The feasibility of TDRP for various biofilm systems was proven by three different biological wastewater treatment processes utilizing TDRP media. The trickling filter system achieved high COD removal efficiency ranging from 79.6-90.1% in various organic loading conditions. The SGBR filled with TDRP was also demonstrated at various HRTs and showed good system stability as evidenced by consistent methane production and over 90% COD removal efficiency. In anoxic conditions, the TDRP filter reactor showed excellent nitrogen removal efficiency during the operating time. Moreover, SEM analysis of the TDRP media collected from trickling filter showed the homogeneous and abundant microorganisms on every surface of TDRP. Therefore, the applicability of TDRP as the biofilm attachment media can have broad applications in environmental fields.

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CHAPTER 3. DEVELOPMENT OF BIOFILTER WITH TDRP (TIRE DERIVED RUBBER PARTICLES) MEDIA FOR ODOR REMOVAL

Jaeyoung Park and Timothy G. Ellis
Department of Civil, Construction, and Environmental Engineering,
Iowa State University, Ames, Iowa, 50011 U.S.A.

Introduction

The most common methods for hydrogen sulfide removal are physicochemical processes (Gabriel et al., 2004). Investigation and application of biological processes, however, have been increasing recently, due to high operating costs and by-products associated with chemical methods (Hansen and Rindel, 2000). Among the biological processes, biofilters, biotrickling filters, rotating biological contactors, bioscrubbers, and suspended cell bioreactors are commonly used for hydrogen sulfide removal treatment (Shareefdeen and Singh, 2005). In consideration of these biological odor removal systems, filter media is one of the most critical factors by providing biomass surface to attach and contact with contaminants (Shareefdeen and Singh, 2005).

Among synthetic filter media materials, rubber material has high potential for application as biofiltration media, owing to its physical and chemical properties. Tire-derived rubber particles (TDRP) from Envirotech Systems, Inc. can be a valuable option for odor removal system biotechnology, since they can be easily produced from the reuse market at low cost. Moreover, several bioreactors with TDRP, trickling filters, hybrid SGBR with TDRP, and anoxic TDRP filters, have successful performances, owing to the applicability of TDRP to the biofilter media (Park et al., 2006). The use of rubber particles in the odor

removal system seems to be a viable option because of the economic advantage of reusing discarded tire materials and the high potential of TDRP media for biofilm attachment. The objective of this research was, therefore, to develop a biofilter system containing TDRP filter media for hydrogen sulfide removal.

Materials and Methods

A biofilter system filled with TDRP was operated with a synthetic hydrogen sulfide gas cylinder. A schematic of the laboratory's pilot biofilter is shown in Figure 3-1. The biofilter consisted of a 49-inch high and 46-inch diameter polyethylene tank, a 4-inch polyvinyl chloride (PVC) inlet pipe, and a 3-inch PVC outlet pipe. This reactor was filled with 4-inch perforated, corrugated HDPE pipes on the bottom of the reactor for sustaining the media bed and venting the treated air. Above the HDPE pipes, three different types of TDRP media were used as the filter bed for system stability—3.4 ft^3 of chunk rubber, 4.4 ft^3 of shredded rubber, and 5.6 ft^3 of fine rubber—located from the bottom bed to the top bed, respectively. Total empty bed volume of this reactor was 13.4 ft^3. The filter media was seeded with 2.1 ft^3 of biomass from the Boone Water Pollution Control Plant.
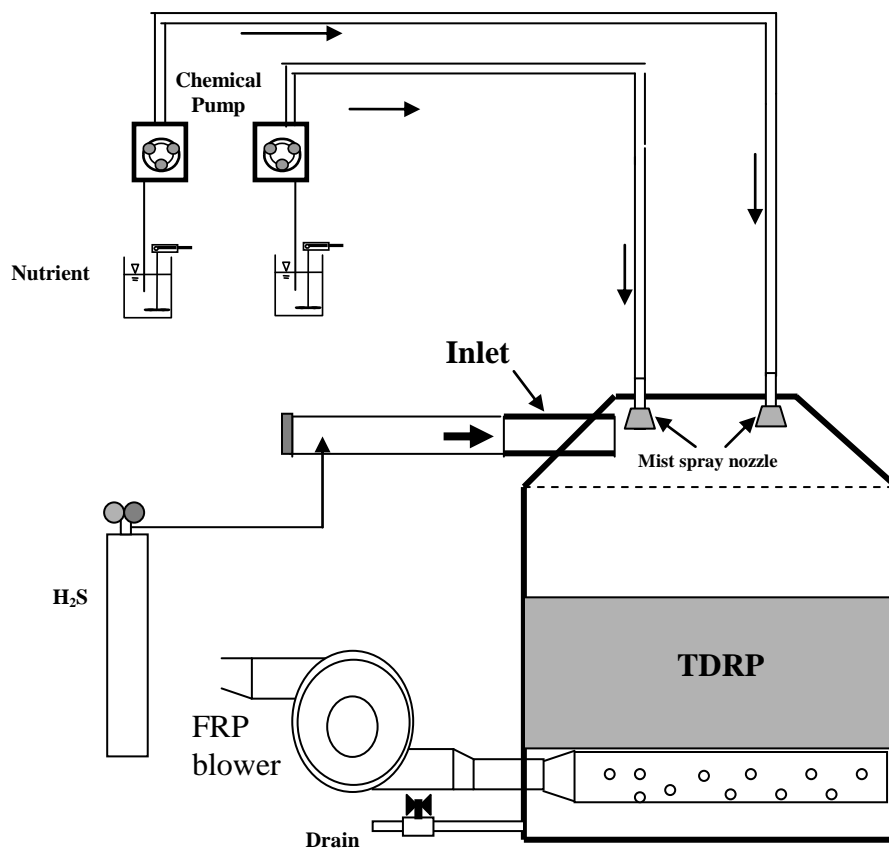
Figure 3-1. Schematic of pilot-scale TDRP biofilter setup

Synthetic hydrogen sulfide was supplied to the main air stream inlet pipeline at the target concentration simulating contaminated air to the pilot reactor. Two spray mist nozzles, a fogging nozzle, and a high-volume clog-resistant misting nozzle were located below the top of the reactor to provide effective moisture content and nutrients to the biomass. A nutrient solution was fed to the reactor by spray mist nozzles at an average flow rate of 1.2 L/h. The constituents of concentrated nutrient solution and trace element solution are shown in Tables 3-1 and 3-2. The concentrated nutrient solution was diluted with tap water by a factor of 10 after the addition of non-fat dry milk concentrated at 0.5g/L.

Table 3-1. Composition of nutrient solution

Compounds	Concentration (g/L)
$NaH_2PO_4H_2O$	0.017
K_2HPO_4	0.02
NH_4Cl	0.11
$MgSO_4.7H_2O$	0.05g
$CaCl_2.2H_2O$	0.005g
Yeast extract	0.003 g
Trace mineral solution	0.07 mL

Table 3-2. Trace mineral solution

Compound	Concentration	Compound	Concentration
$FeCl_2.4H_2O$	10 g/L	$AlCl_3.6H_2O$	0.09 g/L
$CoCl_2.6H_2O$	2 g/L	H_3BO_3	0.05 g/L
EDTA	1 g/L	$ZnCl_2$	0.05 g/L
$MnCl_2.4H_2O$	0.5 g/L	$(NH_4)_6Mo_7O_{24}.4H_2O$	0.05 g/L
Resazurin	0.2 g/L	$CaCl_2.2H_2O$	0.038 g/L
$NiCl_2.6H_2O$	0.142 g/L	HCl (37%)	1 mL/L
Na_2SeO_3	0.123 g/L		

Inlet and outlet H_2S concentrations were measured using a BW Defender multi-gas detector (BW technology, Pantego, TX) at concentration ranging from 0 to 150 ppm. Air flow was measured using a Model 9880 air velocity meter (Terra Universal, Inc., Anaheim, CA). Two water filled manometers were installed at each inlet and outlet line to monitor the pressure change in the system.

Results and Discussion

A gas cylinder having 0.5 - 5 % H_2S and 5% methane was used with a two-stage gas regulator to give the biofilter system consistent H_2S gas concentrations under varying flow conditions. Figure 3-2 shows the performance of the biofilter supplied with H_2S from the gas cylinder. After beginning with the H_2S supply system, the biofilter was operated with an

empty bed retention time (EBRT) of greater than 60s to evaluate initial system stability and provide a maintenance check. The initial airflow rate was 12 CFM, corresponding to a 67s EBRT. There was no H_2S detected in the outflow during this operating condition. A majority of the reported biofilter systems treating H_2S gas are operated below 60 seconds of EBRT, since the biodegradability of sulfide is rapid (Sublette and Sylvester, 1987; Potivichayanon et al., 2005; Wolstenholme and Schafer, 2005). Due to this, the airflow rate of this system was gradually increased to 40.6 CFM (EBRT of 20 seconds) to determine the optimum operating capacity of the TDRP biofilter. The inflow H_2S gas concentration was varied from 20 to 60 ppm at each flow rate in this study. During the various operating periods, there was no H_2S detected (e.g., MDL (minimum detection limit) =1 ppm) in the outflow, indicative of effective H_2S removal performed by the TDRP biofilter system.
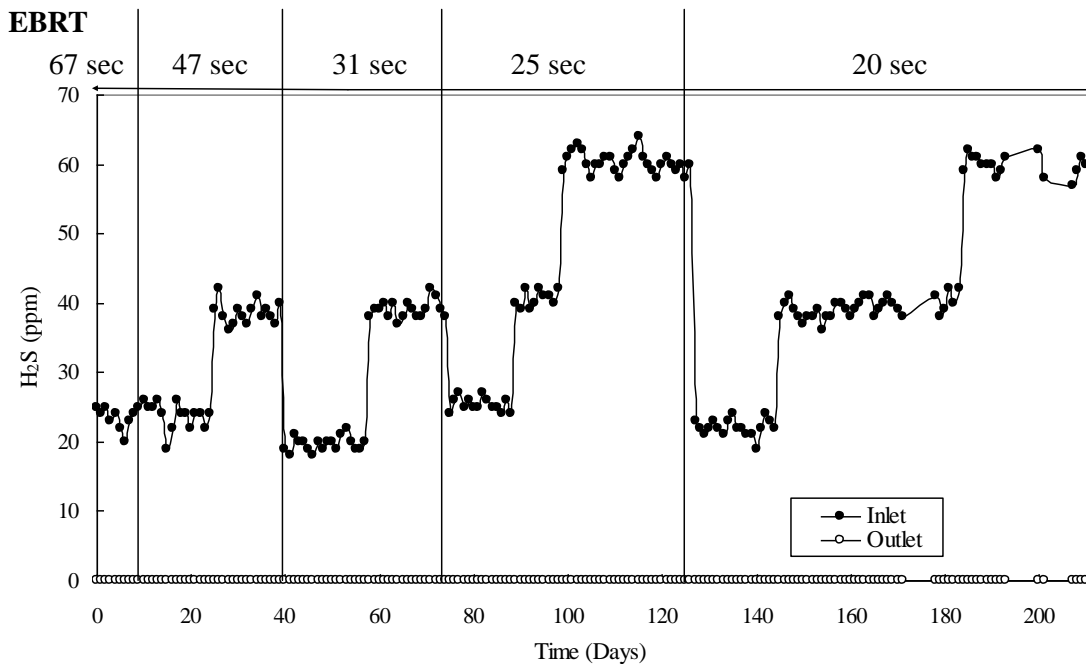
Figure 3-2. Performance of TDRP biofilter system treating H_2S
(Day 0 corresponds to April 24, 2007)

The gas cylinder with 8 % of hydrogen sulfide was replaced for the application before adding higher concentrations of odorous compounds to this system. However, there was a delay in replacement of the gas cylinder, halting hydrogen sulfide supply for 2 months. After replacement, the system was operated at 20-90 ppm of H_2S in inflow with 20-25 seconds of EBRT as shown in Figure 3-3. H_2S was detected in the outlet at a concentration, ranging from 1 to 5 ppm, while the inlet H_2S concentration was above 70 ppm. The removal efficiency decreased at increased H_2S loading rates; although, over 94% H_2S removal efficiency was sustained during the operation, which included over 90 ppm of H_2S inlet concentration.
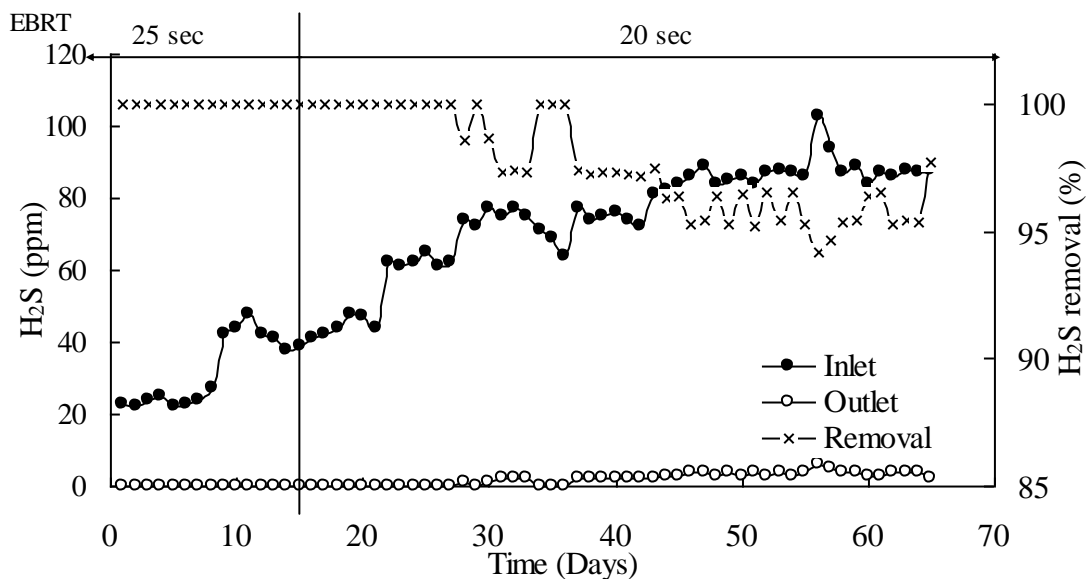
Figure3-3. Variation of H_2S in inlet and outlet with removal efficiency as a function of time (Day 0 corresponds to February 15, 2008)

Inlet mass load was calculated using the equation below:

Inlet Mass load = $\dfrac{Q \times C_{in}}{Vr}$ (3.1)

Where, Inlet Mass load: $g/m^3/hour$

Q: flow rate of inlet (m^3/h)

C_{in}: pollutant concentration in inflow(g/m^3)

Vr : Reactor volume (m^3)
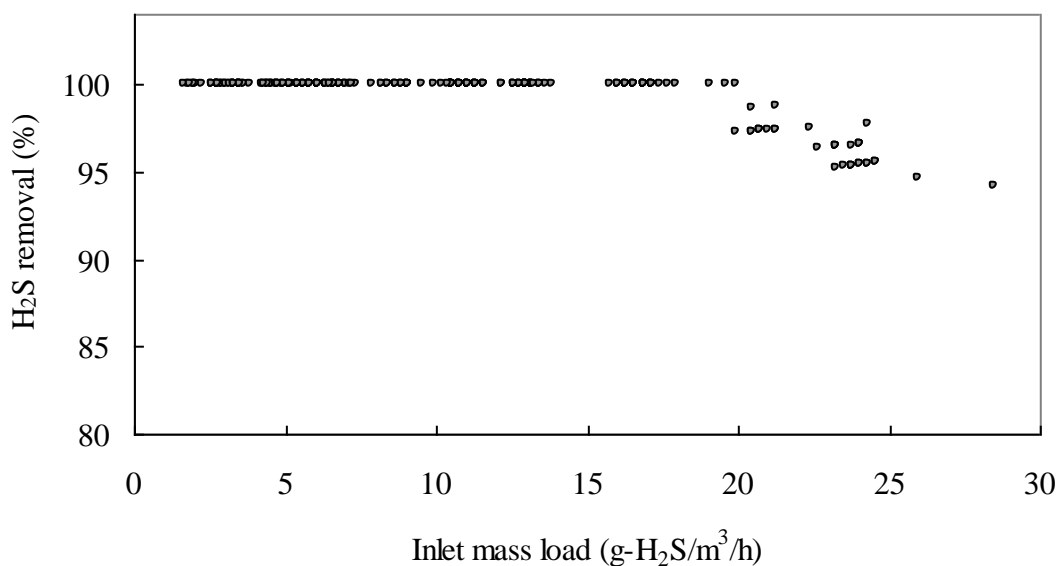
Figure 3-4. The relationship between H₂S removal and mass load

Inlet mass loads were varied from 1.6 to 28.5 g $H_2S/m^3/hour$ during the operation. A majority of the loaded H_2S to the system was removed below 19.6 g $H_2S/m^3/hour$ (Figure 3-4). The H_2S removal efficiency decreased with the mass load above 19.6 g H_2S/ m^3 /hour.

However, the overall system performance was maintained over 94% H$_2$S removal efficiency at the mass loading rates ranging from 19.6 to 28.5 g H$_2$S/m^3/hour.

A common way to evaluate the system performance is by analysis of elimination capacity, calculated as follows:

$$\text{Elimination Capacity} = \frac{Q \times (C_{in} - C_{out})}{Vr}$$ (3.2)

Where, Elimination Capacity: g H$_2$S/ m^3 /hour

C_{out} : pollutant concentration in outflow(g/m^3)

In the odor control system, one of the most important observations is the relationship between the H$_2$S inlet mass load to the system and the elimination capacity of system, owing to its effective indication of system capacity. Normally, increasing the loading rates increases the elimination capacity (Shareefdeen and Singh, 2005). However, this increase of elimination capacity does not continue above certain values of load rates. This is reflected as the maximum elimination capacity (Koe and Yang, 2000). The maximum elimination capacity can be determined at the start of the flat line on the elimination capacity curve, corresponding to the mass load rates (Koe and Yang, 2000). This study shows the increase of elimination capacity along with an increase of mass load rates (Figure 3-5). The maximum elimination capacity was not determined because this plot had no flat line. Therefore, the maximum elimination capacity could be above 25 g-H$_2$S/ m^3 /hour, which

indicates the system could treat H_2S efficiently at loading rates of higher than 25 g-H_2S/ m^3 /hour.
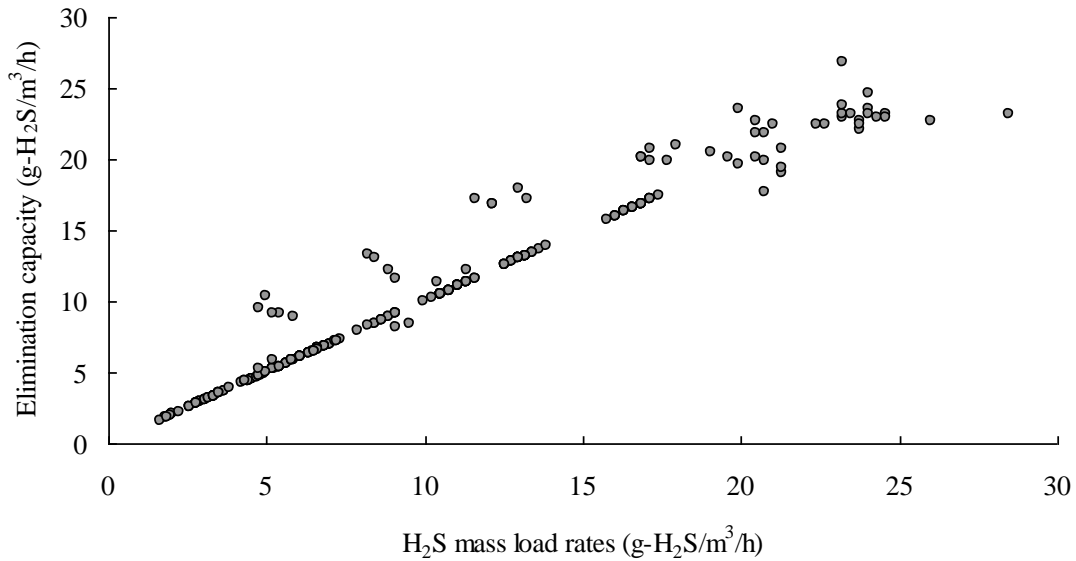
Figure 3-5. The relationship between H_2S load rates and elimination capacity

Conclusions

A biofilter system filled with TDRP media was applied to hydrogen sulfide treatment to evaluate the treatability of the system. During the study, synthetic hydrogen sulfide from the gas cylinder was provided to the TDRP biofilter 20-90 ppm of H_2S in 20-67 seconds of EBRTs conditions. The bioreactor system achieved over 94% removal efficiency of H_2S, which indicates the effective performance of the TDRP bioreactor. This was true as long as EBRTs were not shorter than 20 seconds at less than 100 ppm of H_2S in inflow. Moreover, the maximum elimination capacity of this system was above 25 g-H_2S/ m^3 /hour. Therefore,

this study showed the high potential of this system for hydrogen sulfide removal, owing to

system stability and effective removal efficiency.

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CHAPTER 4. PERFORMANCE COMPARISON OF THE SGBR AND THE ADDITION OF TIRE DERIVED RUBBER PARTICLES (TDRP) TO THE SGBR SYSTEM

Jaeyoung Park and Timothy G. Ellis
Department of Civil, Construction, and Environmental Engineering,
Iowa State University, Ames, Iowa, 50011 U.S.A.

Introduction

Billions of used tires are stockpiled as a result of a shortage of alternatives for reuse scrap tire. There are several alternative applications of used tires to environmental applications such as adsorption materials or filtration media (Mui et al., 2004; Xie and Chen, 2004; Tang et al., 2006). Moreover, the evaluation of tire materials as suitable media for biological growth and biofilm has been performed as an alternative way to reuse the scrap tire besides conventional tire reuses (Shin et al., 1999; Park et al., 2006).

The tire-derived rubber particles (TDRP) from Envirotech system, Inc. showed the potential for various biofilter media in a previous study (Park et al., 2006). Practical application of TDRP to current treatment systems, however, is needed to evaluate it as a potential substitute for developed biofilm. A new anaerobic biological process, called SGBR (static granular bed reactor) was developed at Iowa State University. The SGBR is a simple, down flow anaerobic process using granular sludge (Mach and Ellis, 2000). The comparison of SGBR operation with and without the addition of TDRP could validate the substitution availability of TDRP media for the granule. Therefore, the aim of this study was to determine

the suitability of TDRP as an anaerobic support media. This would help offset the high cost

of anaerobic granules (which traditionally have sold for approximately $66/m^3$).

Materials and Methods

SGBR systems having different compositions of TDRP and granules were tested for

this study. One laboratory scale SGBR reactor and three hybrid SGBR reactors with

different TDRP additions were operated under different hydraulic retention times and organic

loading rates to compare its performance at ambient conditions. Three 1-L active volume

hybrid SGBR reactors were filled with anaerobic granules and TDRP at various proportions

(Figure 4-1). A 2 L active volume SGBR reactor was filled with only anaerobic granules to

compare the performance of TDRP media to the granule. Anaerobic granular seed sludge,

used in the SGBR system, was obtained from the Biothane anaerobic pretreatment unit at the

Cedar Rapids Water Pollution Control Facility in Cedar Rapids, Iowa. These four reactors

were fed from the same synthetic wastewater source, which contained non-fat dry milk.

Experimental conditions of hydraulic retention times and organic loading rates were changed

after the stable operating performance of each reactor was achieved.
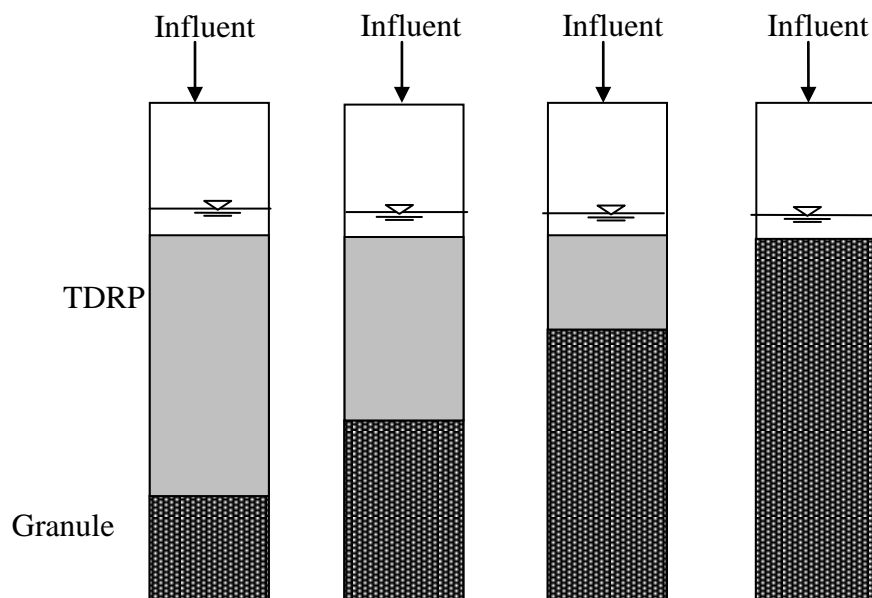
Figure 4-1. Schematics of SGBR and hybrid SGBR reactors with TDRP addition

At each loading condition, chemical oxygen demand (COD), volatile suspended solids (VSS), total suspended solids (TSS), pH, alkalinity, and volatile fatty acids (VFA) were measured, using Standard Methods (APHA et al., 1998) to determine the system's performance. Chemical oxygen demand was measured using the closed reflux, titrimetric method with borosilicate culture tubes. TSS and VSS were measured using Whatman GF/C glass microfiber filters. The distillation method was used for measuring volatile fatty acids. Gas production for each reactor was determined by using a tipping gas meter, while the composition was analyzed using gas chromatography (Gow Mac Instrument Company Series 350 Thermal Conductivity Detector).

Results and Discussion

Initially, a 1 kg/m^3/d organic loading rate was applied to each system, and various organic loading rates were applied, owing to the variation of the hydraulic retention times and organic compound concentration in the feed solution. All operated reactors treated the synthetic wastewater influent under stable conditions, owing to stable organic removal efficiency and low VFA concentration in effluent (Table 4-1). The organic loading rate increased from 1 kg/m^3/d to 4 kg/m^3/d due to the HRT reduction ranging from 48 to 12 hours. However, effluent characteristics from each reactor, were consistent and maintained acceptable qualities during operation. COD concentration in the effluent of each reactor was below 60 mg/L at all HRT conditions. Average effluent TSS and VSS concentrations remained around 30 mg/L each. Produced biogas had 78- 84% methane composition in each reactor.

Table 4-1. Characteristics of influent and effluent from each reactor

	Influent	Effluent			
		R1	R2	R3	R4
COD (mg/L)	1952±52	59.9±4.9	52.8±4.5	46.3±3.0	41.2±4.3
COD removal (%)	-	96.9±0.3	97.3±0.5	97.6±0.2	97.7±0.4
TSS (mg/L)	202±12	32±5	34±4	32±6	33±6
VSS (mg/L)	190±14	28±5	30±6	28±7	29±6
pH	7.34±0.14	7.46±0.11	7.59±0.14	7.57±0.15	7.51±0.15
VFAs (mg/L as HAc)	100±18	9.2±1.7	11.3±2.4	10.5±1.8	9.7±2.4
Alkalinity (mg/L as CaCO$_3$)	575±28	679±41	680±24	686±21	698±24

Hybrid reactors (R1, R2, and R3) treated organic matter in a similar performance to SGBR (R4) during the operation (Figure 4-2). COD concentration in effluent from R1 was

slightly higher than the other reactors. Moreover, SGBR without TDRP addition performed the highest quality of effluent consistently, compared to other hybrid SGBR reactors, owing to lowest COD concentrations in effluent. However, differences among the reactors were small compared to influent COD loading.
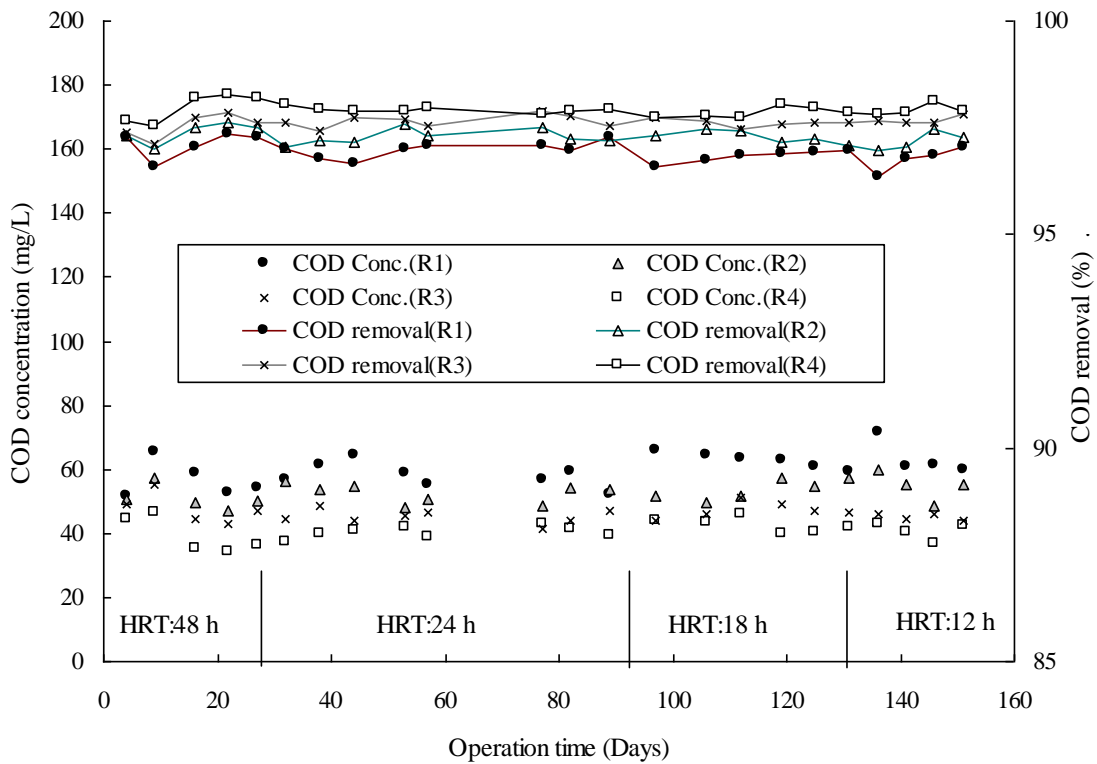
Figure 4-2. Variation of COD in effluent and COD removal of each reactor

COD removal of each reactor was evaluated with statistical analysis using the least significant difference (LSD). The difference between two samples characteristics could be observed by LSD due to declaration of the corresponding population means different (Ryman, 2001). LSD is calculated by the following equation:

$$\text{LSD} = t_{\alpha/2}\sqrt{\frac{s^2 w \times 2}{n}} \tag{4.1}$$

Where, $t_{\alpha/2}$ = student t at α

$s^2 w$ = mean square of within groups

n = number of groups

A student t value and difference between means of COD removal of each reactor were simulated by "Jump," a statistical analyzing program, as shown in Appendix C. LSD of COD removal among the reactors was 0.1169. Table 4-2 shows the differences between COD removals for each reactor and the least squared means. Since the calculated LSD was less than the difference between the means of each reactor, COD removal for each reactor was significantly different in this statistical analysis. Moreover, R4 least squared means was the highest value over R1, R2 and R3, due to the larger volume of granules in the system, indicating granules performed better treatability than TDRP filter media. However, COD removal efficiency for each reactor was held stably near 97% for all HRTs, which affirms the suggested capability of TDRP as a substitute for anaerobic granules in the SGBR system.

Table 4-2. Mean COD removal differences among reactors and least square means

Mean[i]-Mean[j]	R1	R2	R3	R4	Least Sq Mean
R1	0	-0.3596	-0.6957	-0.9796	96.932174
R2	0.35957	0	-0.3361	-0.62	97.291739
R3	0.69565	0.33609	0	-0.2839	97.627826
R4	0.97957	0.62	0.28391	0	97.911739

An additional study of SGBR and hybrid reactor operations was performed to verify the role of TDRP media in the treatment system. The 1L active volume hybrid SGBR (R5) was filled with 0.75 L of TDRP media and 0.25 L anaerobic granule. The performance of this hybrid SGBR (R5) was compared with the operation of 1L active volume SGBR (R6) having the same amount of granular bed volume as the hybrid reactor, 0.25 L. Both of reactors were fed from the same synthetic wastewater source, which contained non-fat dry milk. Overall, COD removal efficiency from the TDRP hybrid SGBR reactor (R5) was greater than 95% at both 24 and 12 hours of HRT. The SGBR reactor (R6) with 25% working volume had poor effluent quality as compared to R5 (Figure 4-3). R6 also performed over 90% of COD removal at 24 hours of HRT. However, R5 had better COD removal efficiency than R6 at this condition and the differences between R5 and R6 increased at higher organic loadings, owing to the rapid drop of COD removal from R6. The addition of TDRP media seemed to provide an increase of treatability to the system, due to active volume increase.
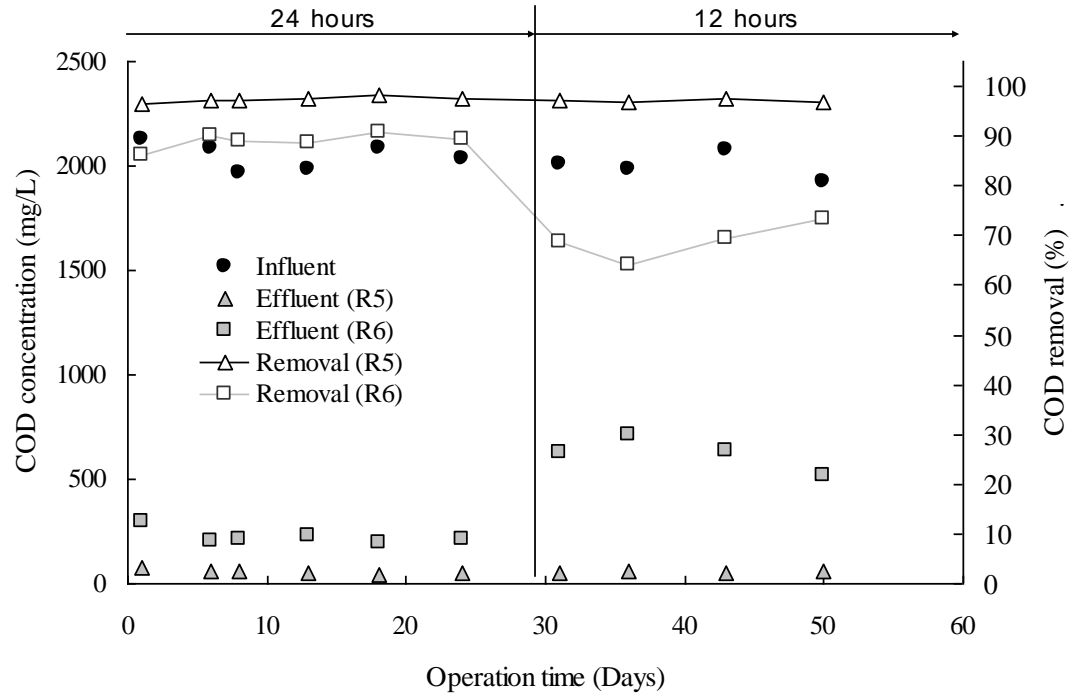
Figure 4-3. COD variations and removal of SGBR and hybrid reactor

Conclusions

A comparison study to evaluate the TDRP media, as a substitute for anaerobic granules, was performed. R1, R2, R3, and SGBR reactors (R4) did not show distinct differences in treatability at various organic loading rates ranging from 1 kg/m^3/d to 4 kg/m^3/d, which indicates applicability of TDRP media to the bioreactor. The differences in organic removal performance between the R5 reactor and the R6 reactor explained the effectiveness of TDRP addition to SGBR system. As a result, the TDRP addition to the

SGBR can provide cost saving for system installation, owing to the economic advantage of

TDRP, and still maintain effluent quality. Despite this, various wastewater application tests

are still required for the TDRP hybrid system to establish operational stability for different

wastewater compositions.

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CHAPTER 5. ON-SITE PILOT DEMONSTRATION OF THE STATIC GRANULAR BED REACTOR (SGBR)

Jaeyoung Park and Timothy G. Ellis
Department of Civil, Construction, and Environmental Engineering,
Iowa State University, Ames, Iowa, 50011 U.S.A.

Introduction

Meat processing plants consume large volumes of water for processing food and washing equipment, which have environmental significance owing to the effluent containing a high concentration of organic matter (Del Pozo et al., 2000; Beux et al., 2007). The typical consumption of water for a slaughterhouse varies from 0.8 to 16.7 m^3/ton live weight in the U.S. and comprises 80% of the fresh water input (Tritt and Schuchardt, 1992; Johns, 1995). Most of the consumed water from a slaughterhouse is discharged as wastewater, including high amounts of organic matter ranging from 4.7 to 9.9kg BOD_5 per slaughtered animal in the U.S. with 40-60% of insoluble fraction (Sayed et al., 1987; Johns, 1995). The insoluble fraction in forms of protein, fats, and cellulose can be degraded slowly and affect the bioreactor performance adversely (Núñez and Martínez, 1999). Therefore, a variety of wastewater treatment systems has been investigated to accomplish appropriate treatments of wastewater generated in slaughterhouses (Tritt and Schuchardt, 1992; Johns, 1995; Caixeta et al., 2002).

Aerobic treatment is not usually considered as appropriate for high strength wastewater such as slaughterhouse wastewater. This is because of the high energy cost

associated with aeration, high solids production rate, and limitation of the oxygen transfer capacity (Torkian et al., 2003). Therefore, anaerobic processes have been considered superior to aerobic systems and have been studied for treatment of complex wastewaters like slaughterhouse wastewater. The effectiveness under high organic loading, energy savings from methane production, significantly lower operating costs, and low sludge production compared to aerobic systems highlight the advantages of anaerobic treatment (John, 1995; Sayed et al., 1988; Chavez et al., 2005). Sayed et al. (1993) reported high treatment performance for a staged UASB (up flow anaerobic sludge blanket) system on slaughterhouse wastewater because of its better sludge stabilization and high organic matter removal efficiency compared to the single stage UASB system (Sayed et al., 1987; Sayed and de Zeeuw, 1998). Moreover, several high rate anaerobic treatment systems such as UASB, EGSB (Expanded granular sludge bed) and AF (anaerobic filter) have been studied for the treatment of food processing wastewater, due to their effectiveness in removing organic matter and their economic advantage compared to the aerobic systems (Harrison et al., 1991; Núñez and Martínez, 1999; Manjunath et al., 2000; Del Nery et al., 2008).

A new anaerobic treatment system called the static granular bed reactor (SGBR) was developed by Ellis and Mach at Iowa State University in the Department of Civil, Construction, and Environmental Engineering (U.S. Patent #: 6,709,591). The SGBR has been successfully demonstrated to treat a variety of wastewaters, including slaughterhouse wastewater from the Hormel Foods Corporation in laboratory and pilot studies performed (Roth and Ellis, 2004). To develop the full-scale design parameters, e.g., hydraulic retention time (HRT), organic loading rate (OLR), and backwash criteria, the system needs to be

demonstrated on a large scale to collect experimental data at various conditions. The repeated performance of the SGBR on a pilot-scale will be a potential guideline for the commercialization of this new technology. The objective of this study was to demonstrate an on-site pilot-scale SGBR system treating slaughterhouse wastewater from a food company in Iowa to provide treatability compared to other high-rate anaerobic systems and critical elements for commercialization.

Materials and Methods

On site pilot scale SGBR system

A 1,000 gallon pilot-scale anaerobic reactor constructed of polypropylene was installed on site at a pork slaughterhouse in Iowa. The on-site pilot scale SGBR reactor consisted of a 1000-gallon reactor with 500 gallons of working volume, an 800-gallon tank for feed storage, 65-gallon tank for effluent storage, 3/4-inch PVC piping and fittings, Masterflex peristaltic pumps (Models L/S 77521-40) and a gas meter (Figure 5-1). A 3/4-inch perforated PVC pipe was placed on the top to distribute the influent across the cross-section the reactor. The underdrain was partitioned by placing four perforated PVC pipes in the middle of the gravel bed within each separate section for effluent discharge and backwashing of the selected section. A semi circular shaped pipe with a 3-inch diameter was installed above the operating water level in the reactor to allow separation and drainage of backwashed water from the granular bed.

The gas produced from the SGBR system was collected through the port on the top and vented to the outside of the building using a 3/4-inch PVC piping. The biogas production rate during the system operation was measured using a Schlumberger oil gas meter after passing through a steel wool scrubber to remove hydrogen sulfide. The manual control of several valves connected to the gas venting system prevented a sudden rise of pressure in the pilot reactor during backwashing. The pressure change in the reactor was monitored using a manometer.
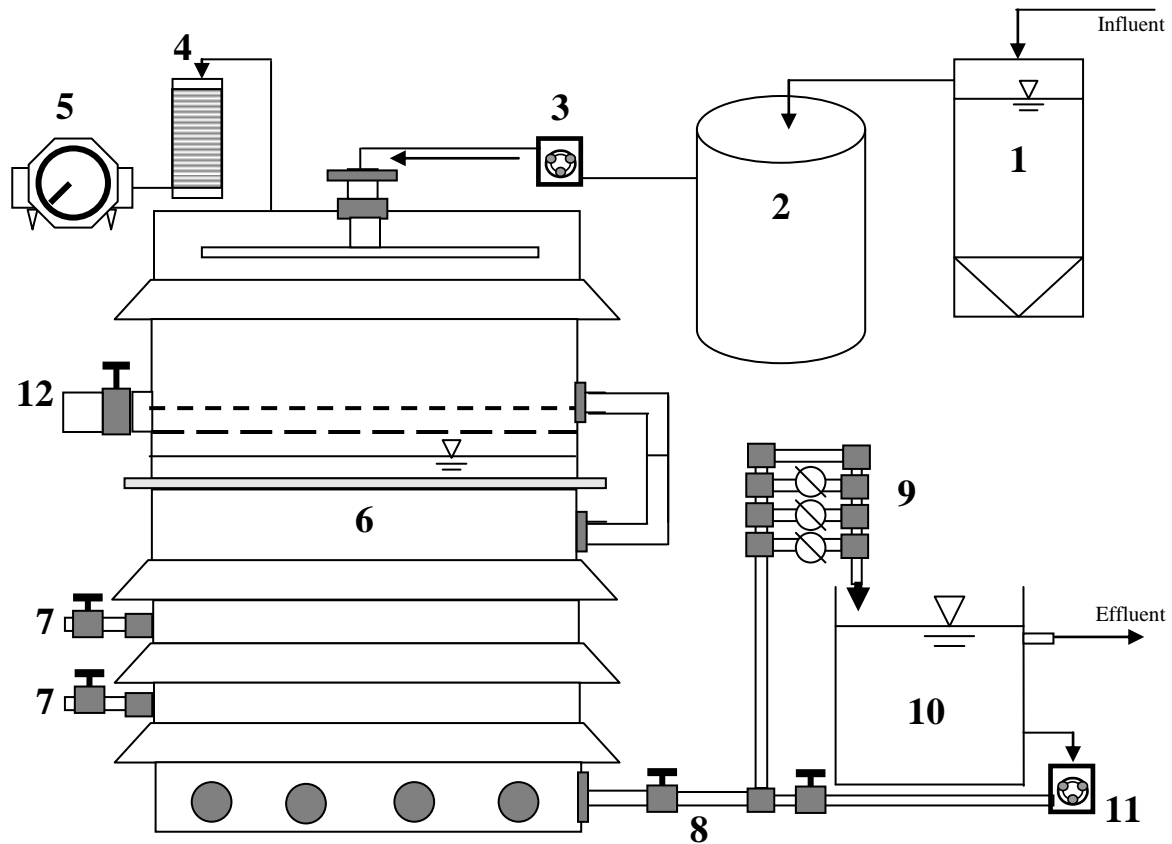
Figure 5-1. Schematic of the pilot-scale SGBR system; 1, DAF tank; 2, Influent storage tank; 3, Influent pump; 4, H₂S scrubber system; 5, Gas meter; 6 SGBR reactor; 7, Sampling port; 8, Drain and backwashing valve system; 9, Effluent overflow pipe; 10 Effluent storage tank; 11, Backwash pump; 12, Backwashing water discharge system

The reactor was seeded with approximately 400 gallons of anaerobic granules from City Brew Brewery in La Crosse, Wisconsin. The reactor was fed with wastewater from the dissolved air flotation (DAF) unit at the food processing plant. Treated effluent from the reactor was discharged with the DAF effluent to the onsite lagoon treatment system. The SGBR system was operated on a continuous basis for four-day HRT conditions initially to maintain 1 kg/m^3/d of organic loading rate. The hydraulic retention time (HRT) and organic loading rate (OLR) were varied after the start-up to demonstrate the feasibility of the SGBR system applied to the slaughterhouse wastewater. The planned range of operating HRTs was 20 to 48 hours. After steady-state conditions were observed for a period of time, changes in hydraulic retention time and organic loading rate were initiated by changing the influent feed rate.

Analytical methods

A portion of the influent and effluent was sampled and analyzed three times a week to monitor the performance of the reactor. For this analysis, chemical oxygen demand (COD), biochemical oxygen demand (BOD), volatile fatty acids (VFAs), total suspended solids (TSS), and volatile suspended solids (VSS) were determined according to Standard Methods for the Examination of Water and Wastewater (APHA,1998). The COD tests and VFA tests were performed by the closed-reflux method and distillation method, respectively. TSS and VSS measurements were performed by the filtration method (Standard Methods, section 2540 D and E) with glass fiber filter paper (Whatman GF/C, 1.2 μm pore size). The soluble COD was also measured using the filtered sample wastewater to monitor the mass balance in

the system. The influent and effluent wastewater pH were measured using an electronic pH

meter (Thermo Orion 210A).

Biogas analysis and hydrogen sulfide measurements were performed during the

operating period. Biogas was sampled with a 100 mL glass gas sampler transported to the

ISU analytical laboratory and analyzed with a Gow Mac gas chromatograph for gas

composition. Hydrogen sulfide was measured, using Dräger short-term measurements H_2S

detector tubes.

SMA (Specific methanogenic activity) test

The SMA tests were performed in batch tests during the study to evaluate the activity

state of biomass employed in the pilot scale system. The batch test applied to this study was

the modified SMA tests used by Rinzema et al. (1988). A 250 mL glass serum bottle was

used for the batch reactor sealed by a rubber septum. All tests were performed in duplicate at

constant temperature (35 ^{o}C) and 160 rpm oscillations controlled in a shaker (Incubator

shaker series 2, New Brunswick Scientific Co., Inc.). Acetic acid was dosed to the serum

bottle with a 0.5 ~ 1.0 F/M ratio to avoid substrate inhibition after the addition of batch

medium solution, buffer solution, and biomass obtained by the pilot SGBR reactor. The head

space of the test bottle was adjusted to approximately 100 mL by the addition of anaerobic

water solution to provide effective gas measurements. The amount of biogas produced was

measured and released by a glass syringe at regular intervals depending on the gas production

rate. The methane concentration increase by the continuous production of biogas in the

headspace of the test bottle was measured using a Gow Mac gas chromatograph. The

methane activity was specified by the mass of produced methane divided by the mass of

biomass and time, expressed by g methane production per g biomass per day. The detailed

test procedure, including a list of the reagents used in the test, is described in Appendix B.

Results and Discussion

Start-up of SGBR reactor

The pilot scale SGBR system was started at 4 days HRT to provide the seeded

biomass sufficient time for acclamation to the slaughterhouse wastewater. At this initial

operation with 5,000 mg COD/L and 2,000 mg TSS/L influent concentration, the COD and

TSS concentrations in effluent averaged 1,100 mg COD/L and 730 mg TSS/L, respectively.

The high organic matter concentration in effluent during the start up period can be explained

by the washout of some anaerobic granule debris. Crushing of the granules may have

occurred during the seeding process. Effluent COD, however, gradually decreased to

300mg/L within 10 days of operation. Effluent TSS and VSS removal also showed gradual

improvements within 10 to 15 days as their effluent values decreased to 96 mg/L and less.

The COD and solids influent concentration values were fluctuated, due to the

variation in slaughterhouse wastewater characteristics. Even the startup operation of the

system at long HRT had various organic loading rates from 0.8 to 2.3 kg/m^3/d, owing to the

variation of influent wastewater strength. Overall COD and TSS removal efficiency were

higher than 95%, which indicated the system was stable at this initial operating condition as

shown in Figure 5-2. After 10 days of operation, the SGBR system treated slaughterhouse

wastewater effectively, due to the rapid acclamation of the system. Therefore, this pilot scale

SGBR system showed high potential to treat slaughterhouse wastewater during the startup

operation.
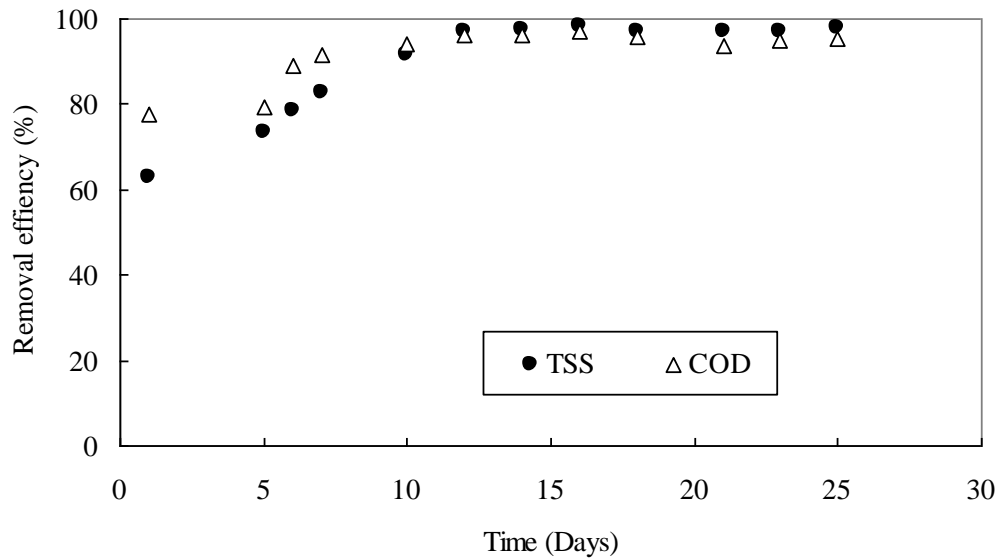
Figure 5-2. The variation of TSS and COD removal efficiency in the SGBR system during initial startup

Overall system operation

This study showed that the SGBR system removed TSS and COD with over 95%

removal efficiency during the operation (Figure 5-3). After the start-up operation under 4 d

HRT, the system was operated at various HRTs ranging from 48 to 20 h to evaluate the

SGBR treatment capabilities for slaughterhouse wastewater. During the HRT decrease from

96 to 48 h, there was no diminishment in treatment performance, primarily because of

consistently low concentrations of organic matter in the effluent. Overall effluent TSS and

VSS concentrations were below 80 mg/L during the 48-hour HRT condition. The effluent

COD concentration was also consistently lower than 300 mg/L during this operating

condition. There was little increase of the effluent COD and TSS concentration just after the

backwashing during the 48 h HRT condition, owing to rapid discharge of retained

wastewater in the system.
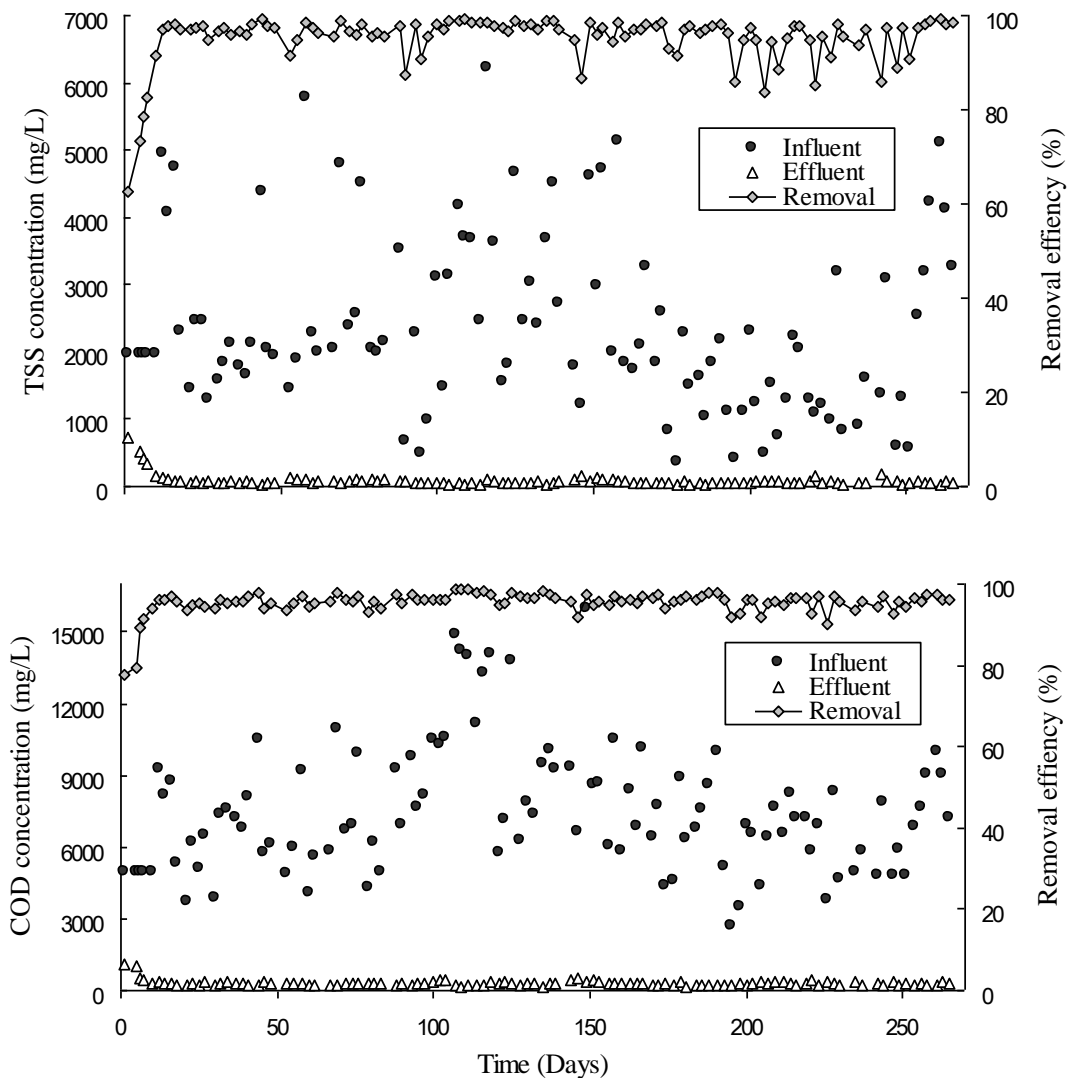
Figure 5-3. Variation of COD and TSS concentrations with removal efficiency in pilot SGBR system

When the flow rate was increased to 330 gal/d, corresponding to a 36 h HRT, the SGBR system was operated at a constant HRT condition from Monday through Friday and a longer HRT (e.g., 48 h) during the weekend because of storage tank capacity. Slaughterhouse wastewater was not generated during the weekend. The SGBR system was operated at a 48 h HRT for a couple of days every week while the system HRTs were 36, 30, 24, and 20 hours. Despite the hydraulic and organic shock loading that the system was subjected to each week when HRT was changed from the 48 h weekend HRT to the weekday HRT, the SGBR maintained consistent performance (Figure 5-3). Even at a turn down ratio of 2.4:1, the SGBR showed no signs of stress or potential upset conditions (e.g., increase in VFA concentration or decrease in COD removal).

The COD and solids removal efficiencies were also consistently higher than 95% at 36, 30, 24, and 20 h HRT (Table 5-2). The average effluent TSS, VSS, COD, soluble COD, and BOD_5 concentrations were 84, 71, 301,197, and 87 mg/L, respectively. The effluent COD values were not as low when compared to other SGBR studies (Mach and Ellis, 2000; Roth and Ellis, 2004; Evans and Ellis, 2005). In consideration of the low value of the ratio of BOD_5 to COD (0.3 or less in this study), however, the majority of the biodegradable organic matter was removed in this system.

Table 5-1. Influent wastewater characteristics

	HRT (hours)						Overall study
	96	48	36	30	24	20	
Operating time (Days)	1-30	31-62	63-132	133-174	177-216	217-265	
TSS (mg/L)	2505 ±1425	2350 ±1208	2786 ±1326	2800 ±1322	1413 ±652	2094 ±1463	2355 ±1321
VSS (mg/L)	2327 ±1399	2194 ±1232	2697 ±1322	2768 ±1364	1333 ±683	1985 ±1418	2255 ±1319
Total COD (mg/L)	5659 ±1753	6773 ±1722	9238 ±3141	8494 ±2598	6556 ±1899	6710 ±1907	7864 ±4294
Soluble COD (mg/L)	3214 ±738	2928 ±483	4033 ±789	3354 ±889	3297 ±855	3519 ±1508	3489 ±985
BOD_5 (mg/L)	NM	NM	NM	6288 ±985	6057 ±965	5571 ±1626	5732 ±1522
VFA (mg/L as HAc)	348 ±94	644 ±88	885 ±296	1286 ±180	1143 ±127	1319 ±167	935 ±385
pH	5.52 ±0.22	5.55 ±0.23	5.74 ±0.36	5.68 ±0.29	5.66 ±0.15	5.64 ±0.28	5.64 ±0.26
Alkalinity (mg/L as $CaCO_3$)	58±24	128 ±36	323 ±202	292 ±139	296 ±81	278 ±104	264 ±157
Organic loading rate (kg $COD/m^3/d$)	1.41 ±0.44	3.39 ±0.86	5.52 ±1.90	6.00 ±2.51	5.47±2.29	6.19 ±3.06	4.84 ±2.59

Table 5-2. Effluent characteristics

	HRT (hours)						Overall study
	96	48	36	30	24	20	
Operating time (Days)	1-30	31-62	63-132	133-174	177-216	217-265	
TSS (mg/L)	203 ±207	67 ±26	63 ±22	78 ±34	56 ±18	68 ±37	84 ±92
TSS removal (%)	92.2 ±7.8	96.7 ±2.0	97.0 ±2.5	96.2 ±2.9	94.8 ±4.3	95.4 ±4.1	95.3 ±5.4
VSS (mg/L)	174 ±184	59 ±22	56 ±21	69 ±33	48 ±16	53 ±16	71 ±81
Total COD (mg/L)	424 ±275	287 ±44	278 ±62	320 ±91	257 ±55	280 ±49	301 ±126
COD removal (%)	92.1 ±5.8	95.6 ±2.1	96.6 ±1.4	96.0 ±1.5	95.7 ±1.8	95.4 ±2.0	95.4 ±2.9
Soluble COD (mg/L)	204 ±24	201 ±43	203 ±54	187 ±56	194 ±33	193 ±35	197 ±45
BOD_5 (mg/L)	NM	NM	NM	77 ±20	88 ±22	90 ±15	87 ±24
VFA (mg/L as HAc)	16 ±4	13 ±2	19±7	19 ±6	21 ±10	19 ±4	18 ±6
pH	6.88 ±0.19	7.19 ±0.3	7.44 ±0.29	7.36 ±0.19	7.25 ±0.2	7.18 ±0.23	7.27 ±0.28
Alkalinity (mg/L as $CaCO_3$)	613 ±43	516 ±80	786 ±114	758 ±125	718 ±103	613 ±43	715 ±132

The biogas production increased along with the increase of the removed organic matter at the shortened HRT. In addition, the rate of increase in the water level in the SGBR system increased as the flow rate increased (and HRT decreased). The hydraulic profile was maintained in the reactor by backwashing the system at regular intervals on an as needed basis.

The influence of the organic loading rate on the system process efficiency was evaluated under diverse organic loading conditions ranging from 0.77 kg/m^3/d to 12.76 kg/m^3/d. This variation of organic loading rates was due to the hydraulic loading variation for system optimization and the frequent fluctuations of COD concentrations in the effluent from the DAF. The COD removal efficiency in SGBR system was consistently 95% or better at the various organic loading rate conditions (Figure 5-4).

The lower COD removal efficiency at the organic loading rates or 1 kg/m^3/d and lower was due to the system's instability during startup. TSS removal efficiency at most organic loading rates was above 90%, except at the organic loading rates of 1 kg/m^3/d and less as shown in Figure 5-5. There was little effect of organic loading rate on COD and TSS removal efficiency. Even at the highest organic loading rate of 12 kg/m^3/d, the system performance maintained high COD and TSS removal efficiencies. Moreover, this pilot-scale SGBR consistently maintained over 90% COD removal efficiency, even during rapid organic loading rate changes.
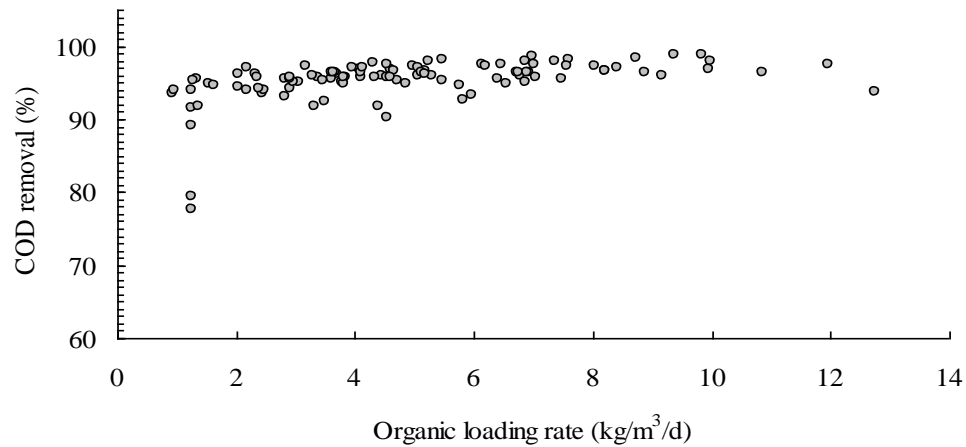
Figure 5-4. COD removal efficiency at various organic loading rates
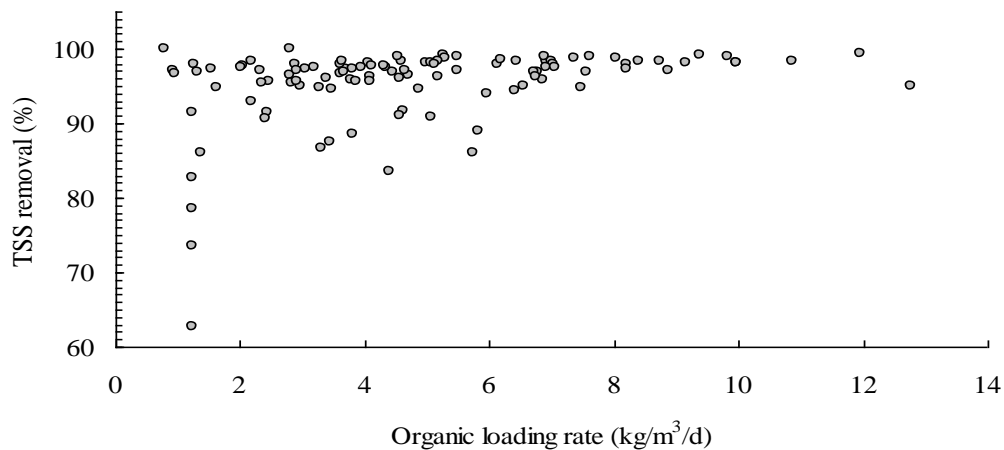
Figure 5-5. TSS removal efficiency at various organic loading rates

Figure 5-6 shows the comparison in COD removal efficiency between the SGBR system and several anaerobic systems treating slaughterhouse wastewater at various organic loading rates. The average COD removal efficiencies at each organic loading rate were plotted for the pilot-scale SGBR system in Figure 5-6. The high COD removal was achieved by the SGBR compared to other anaerobic systems at each organic loading condition.

Among anaerobic systems, only the ASBR (Massé and Masse, 2000) achieved higher COD

removal efficiency than this study at the organic loading rate condition of less than 2

kg/m^3/d. This was due to the lower COD removal by the SGBR's instability during startup.

Overall COD removal efficiency of the SGBR system was greater than other anaerobic

systems, even at higher organic loading rate conditions, indicating the potential for the SGBR

system to treat slaughterhouse wastewater at various organic loading rates.
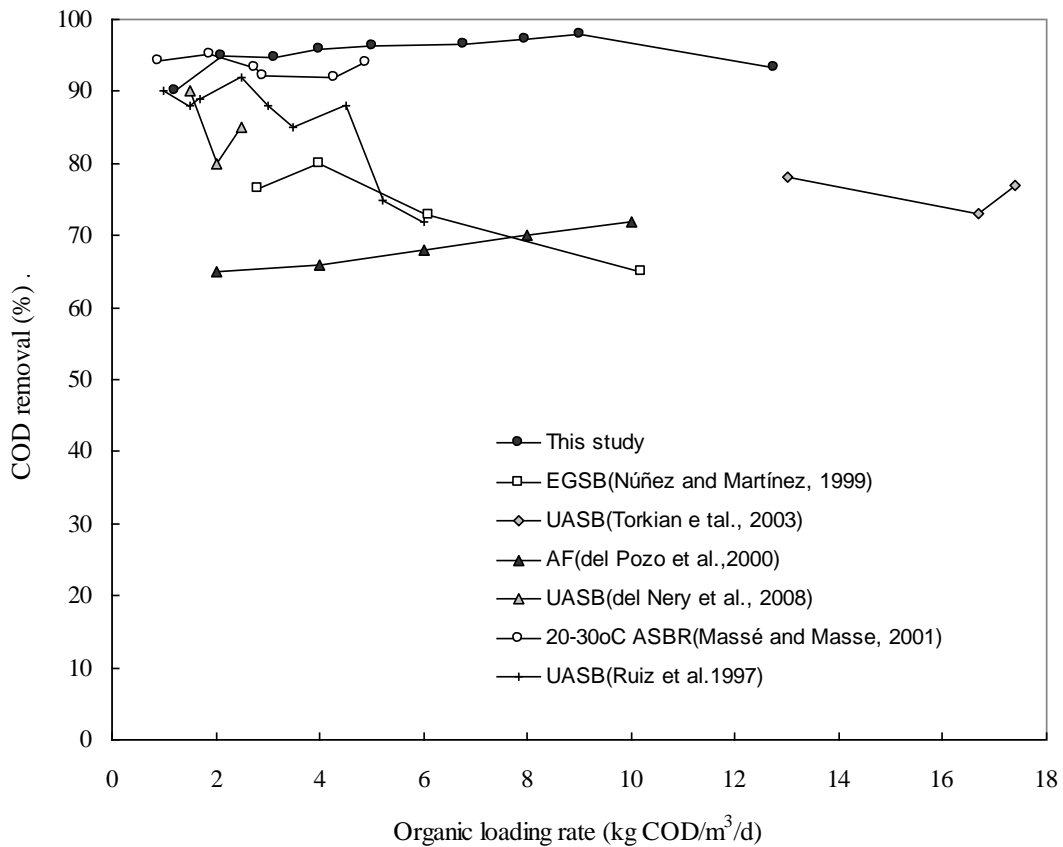
Figure 5-6. Comparison of COD removal efficiency between pilot-scale SGBR and other anaerobic treatment system for slaughterhouse wastewater at various organic loading rates

pH, Alkalinity and VFA

Figure 5-7 showed the variation of pH, alkalinity and VFA in the influent and effluent during operation. Effluent pH, alkalinity, and VFA values did not fluctuate much compared to their influent values due to the stability of the system. Average pH, alkalinity, and VFA in effluent were 7.3, 697 mg/L as $CaCO_3$, and 7.3 mg/L as HAc, respectively. The effluent VFA was less than 30 mg/L as HAc during the operation, which indicates there was no accumulation of fermentation intermediates, such as VFAs providing unfavorable conditions to the anaerobic biosystem. An increase in effluent pH and alkalinity was observed in the system. A similar pH and alkalinity increase was reported often from bioreactors treating slaughterhouse wastewater, due to the high proportion of protein (Massé and Masse, 2000; Roth and Ellis, 2004). The bicarbonate generated by the conversion of protein to ammonia, which provided the additional buffer capacity, caused an increase in alkalinity. The addition of alkalinity to the influent for system stability was not required.
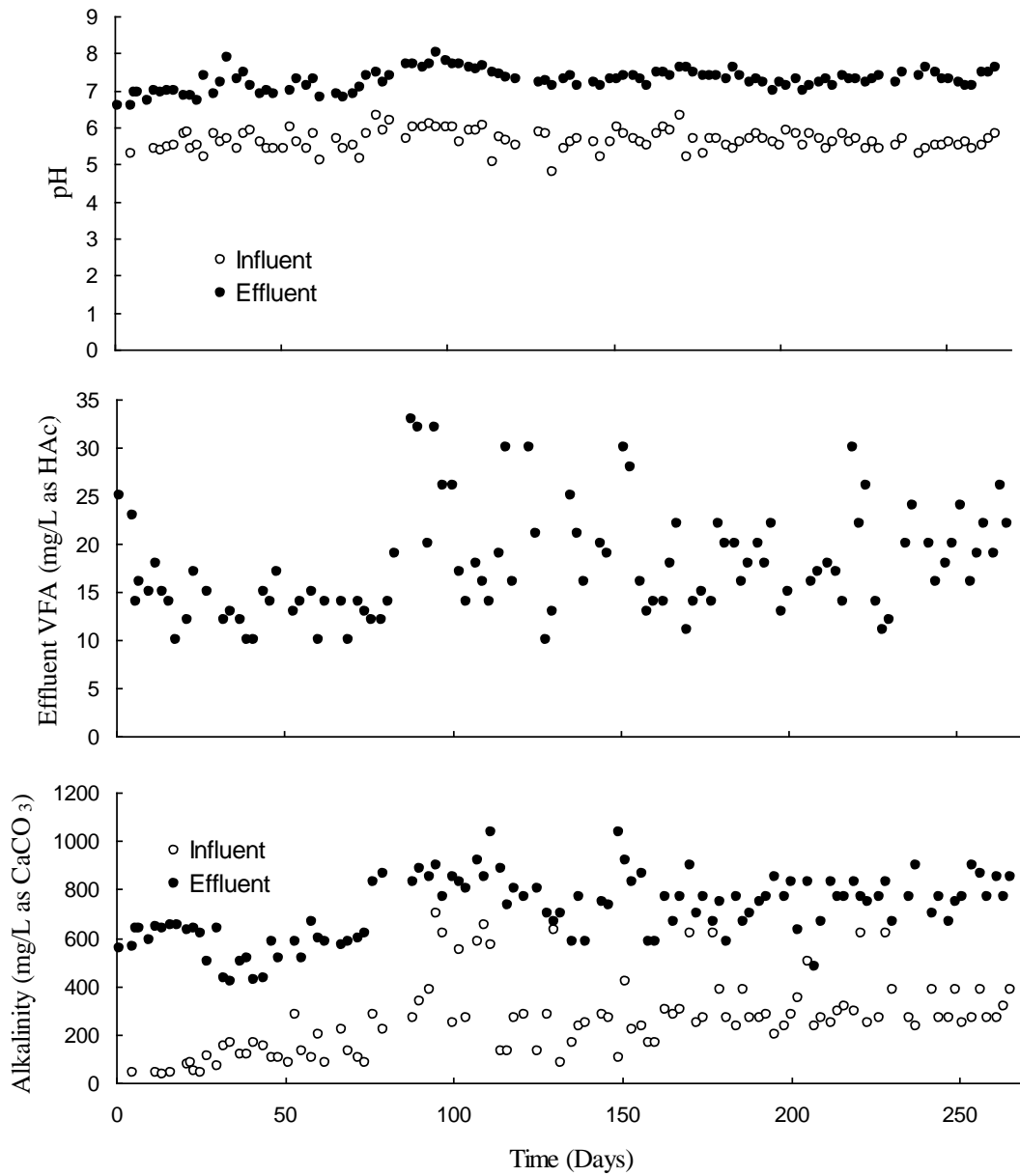
Figure 5-7. Variation of pH, VFA, and alkalinity of pilot-scale SGBR

Solids concentration in granular bed

Solids concentrations in the granular bed were measured to evaluate the variation of biomass concentration in the system during operation. The lab-scale SGBR often showed higher TSS concentration with an increase in granular bed depth. In this study, TSS and VSS concentrations of bottom granules were similar or slightly higher than the sludge in the middle of the granular bed (Figure 5-8). Therefore, there was no serious compression of sludge, which may cause system instability by crushing the granules and disrupting the underdrain system.
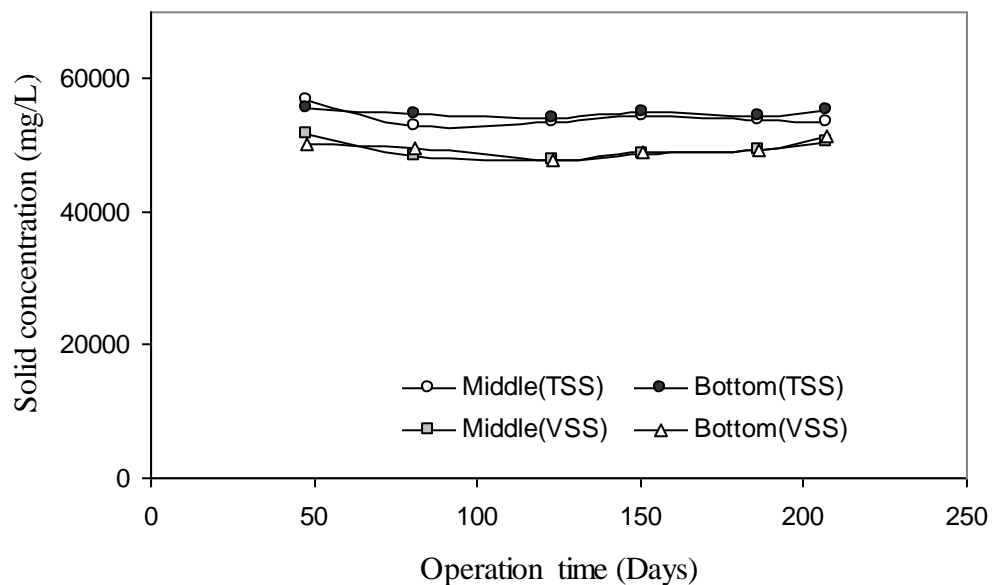
Figure 5-8. Variation of TSS and VSS concentrations in middle and bottom granules

Backwashing

Since the SGBR is a down flow system, increases in head loss occurred frequently, due to the clogging of the underdrain system by the solids accumulation in the reactor. This

problem is alleviated by periodic backwashing performed using the effluent underdrain

system and the collected effluent. Moreover, periodic backwashing can provide a mixing

effect, which alleviates the compression of anaerobic granules and channeling of the flow in

the granular bed.

The backwash was performed for 18 minutes using 65 gallons of collected SGBR

effluent. Four separate bottom drains were used independently and together for passing

backwash water into the SGBR, using the drain valve system (Table 5-3). The first 4 steps in

the backwash performance alleviated the clogging in each section of the SGBR underdrain,

and the last step provided overall separation of the accumulated solids from the granular bed.

Table 5-3. Backwashing working performance at each underdrain valve

	Normal operation	Backwashing working time (minutes)					Drain the backwashed water
Time	-	3.5	3.5	3.5	3.5	4	-
Valve 1	Open	**Open**	Close	Close	Close	**Open**	Close
Valve 2	Open	Close	**Open**	Close	Close	**Open**	Close
Valve 3	Open	Close	Close	**Open**	Close	**Open**	Close
Valve 4	Open	Close	Close	Close	**Open**	**Open**	Close

During the backwash, an equal or excess amount of the effluent used for backwash

was drained, using the wash trough installed above the normal operation water level. TSS

concentration in the drained backwash water was varied at the elapsed time of the drain as

shown in Figure 5-9. After backwashing, TSS concentration in the liquid volume above the

granular bed increased, due to the flotation of solids separated from the granular bed. The

concentrated solids in the liquid volume were discharged by a drain port. After draining, the

TSS concentration was approximately restored to the influent level, which indicated

negligible washout of anaerobic granules by the backwash.
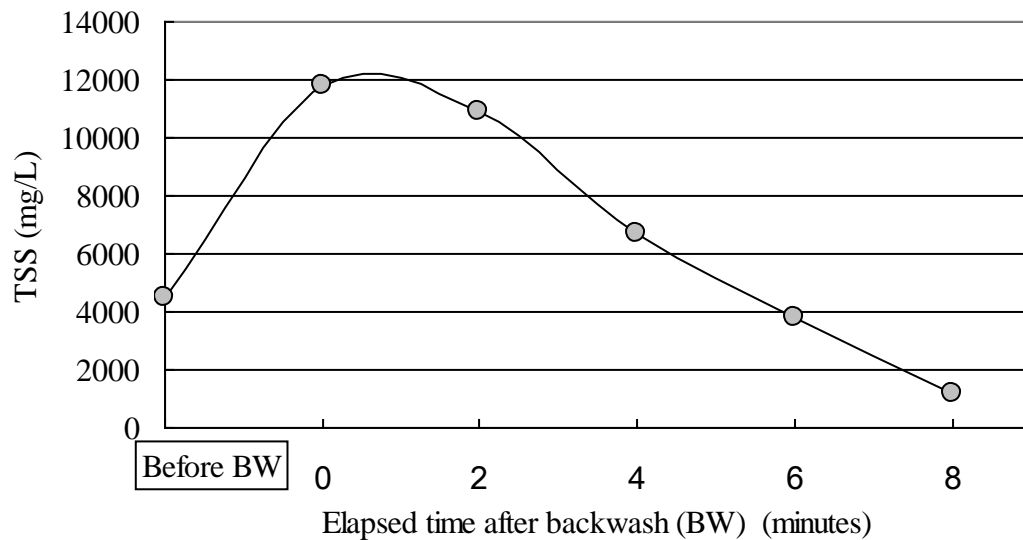
**Figure 5-9. Variation of TSS concentration in drained backwashing water
at elapsed time**

Solids balance on the system

The SGBR treating slaughterhouse wastewater showed over 95% solids removal

efficiency at various solids loading conditions, owing to its ability to act as a biofilter system.

The stored organic solids were degraded by the anaerobic microorganisms in the system.

There was some solids accumulation in the SGBR during the operation due to non-

biodegradable solids and the slow biodegradation of solids compared to the soluble organic

matter. Backwashing was utilized to remove over-accumulated solids and non biodegradable

solids in the SGBR, which maintained the optimum head loss in the effluent discharge system. After backwashing, the solids balance between the accumulated solids in operation and the removed solids by backwashing was evaluated to optimize the system performance as well as the backwashing periods and strategies.

There were several assumptions made to calculate the solids balance on the system. At first, it was assumed that the removed COD was converted to methane with the ratio of 0.35L / g COD for the standard point. Second, the removed solids by methane conversion were approximated by the calculation of methane production divided by the ratio of insoluble COD to TSS as indicated below.

$$TSS_{conv} \text{ (TSS converted to CH}_4\text{)} = \frac{CH_4 \text{ production (L) from TSS}}{0.35 \text{ (L/g COD)} \times \text{(COD insoluble/ TSS)}} \qquad (5.1)$$

$$COD \text{ insoluble} = COD \text{ total} - COD \text{ soluble} \qquad (5.2)$$

Since the influent wastewater characteristics varied consistently, the average values of the ratio of insoluble COD to TSS were calculated and applied to the solids balance evaluation at each backwashing period interval. The methane composition in the biogas produced was measured three days a week. The other four days were averaged with the acquired data as the methane composition in produced gas was stable.

Finally, it was assumed that all soluble COD removed by the SGBR was converted to methane with the theoretical conversion ratio. The difference between total methane production and this calculated methane production by soluble COD removal could represent the value of methane conversion by solids degradation. Therefore, TSS accumulation was calculated as follows.

$$TSS\text{ }_{accumulation}\text{ (kg)} = TSS_{in}\text{ (kg)} - TSS_{out}\text{ (kg)} - TSS_{conv}\text{ (kg)} - TSS_{backwash}\text{ (kg)} \quad (5.3)$$

$$CH_4\text{ from TSS} = \text{Total }CH_4\text{ production} - CH_4\text{ from SCOD removal} \quad (5.4)$$

$$CH_4\text{ from SCOD removal} = (SCOD_{in}(g) - SCOD_{out}(g)) \times 0.35\text{ (L/g COD)} \quad (5.5)$$

The calculation of solids balance was performed at each backwashing period to evaluate the solids accumulation in the system. Table 5-4 shows the overall solids load and discharge for each operating period. The total loaded solids mass varied at each operating period, due to the variation of TSS concentration in the influent. Moreover, the fluctuation of methane production was influenced by the variation in organic loading that occurred by the fluctuation of COD concentration in the influent.

Table 5-4. Solids in and out at each backwashing interval

Backwash interval (Days)	HRT (hours)	Solids loaded by influent (g)	Solids discharged by effluent (g)	Solids remained in SGBR (g)	Total CH$_4$ production (L)
12	48	26,393	879	25,514	19,167
17	36	50,355	1,438	48,917	40,995
17	36	26,629	1,074	25,555	39,468
12	36	41,955	590	41,365	38,171
17	36	59,131	1,075	58,056	44,501
7	36	24,082	422	23,660	18,193
12	30	35,829	1,294	34,535	36,008
12	30	40,826	1,558	39,268	35,941
9	30	26,180	746	25,434	24,928
14	30	32,788	917	31,871	41,431
10	24	25,901	672	25,229	33,800
8	24	16,751	686	16,065	19,294
10	24	13,381	1,078	12,303	23,508
9	24	20,626	715	19,911	28,962
8	20	16,888	1,410	15.478	27,912
7	20	18,162	500	17,662	18,412
9	20	14,851	1,258	13,593	19,420
5	20	11,466	526	10,939	14,301
9	20	33,657	884	32,773	34,360

Table 5-5 shows the variation of the accumulated solids calculated by the addition and subtraction of balanced solids. The removed solids from the discharge of backwashing were varied at each operating period, because of the differences in accumulated solids in the system at each backwash interval. The calculated solids accumulation in the system was positive or negative, due to more or less discharge of the accumulated solids by backwashing. However, there was no serious washout of biomass or significant accumulation of loaded solids at each backwashing interval, which exemplifies the system's stability under consideration of solids accumulation.

Table 5-5. Solids balance at each backwashing interval

Backwash interval (Days)	HRT (hours)	CH$_4$ from solids degradation (m^3)	Solids removed by degradation (g)	Solids removed by backwash (g)	Solids accumulations (g)
12	48	9,329	23,371	2,081	61.8
17	36	21,141	47,265	1,833	- 181.3
17	36	13,479	22,877	2,458	219.8
12	36	16,085	38,298	2,939	126.3
17	36	22,639	53,902	4,321	- 167.8
7	36	8,806	16,378	7,294	- 11.6
12	30	16,241	26,040	8,482	12.1
12	30	23,442	32,224	6,514	529.6
9	30	14,084	19,766	6,160	-492.3
14	30	16,511	24,994	6,683	-36.1
10	24	14,249	19,191	5,807	229.9
8	24	9,120	10,172	3,506	- 129.0
10	24	11,145	11,135	1,242	- 78.3
9	24	14,180	16,000	3,852	57.8
8	20	9,568	12,380	3,247	-149.7
7	20	7,235	14,144	3,379	139
9	20	7,832	11,306	2,357	-70.5
5	20	6,838	9,167	1,854	-82.6
9	20	17,061	27,035	5,504	203

The solids balance and cumulative solids are shown in Figures 5-10 and 5-11. There were several negative values, due to the influence of accumulated solids remaining from previous backwashes. After a large positive value of the remaining accumulated solids appeared, a large negative value for the solids balance was calculated the next turn. This is seen when the remaining solids from the backwash affects the next turn of backwash solids balance calculations, due to the discharge from the current backwash turn.

The cumulative solids amount was positive after Day 24, as a result of the lower removal of solids from the backwash than those retained in the SGBR. Therefore, there did not appear to be serious losses of granular sludge in the SGBR system by washout during

backwashing. Moreover, there was no appearance of significant solids accumulation, owing

to the less than 600 g of cumulative solids maintained during the operation. This indicates no

detrimental effects by the solids accumulation. The cumulative values fluctuated little, due

to the variation of solids balance at each backwash turn. However, the cumulative values

remained less than 300 g, except the 94-106th day of operation, indicative of a stable, solids

balance maintained during this study.
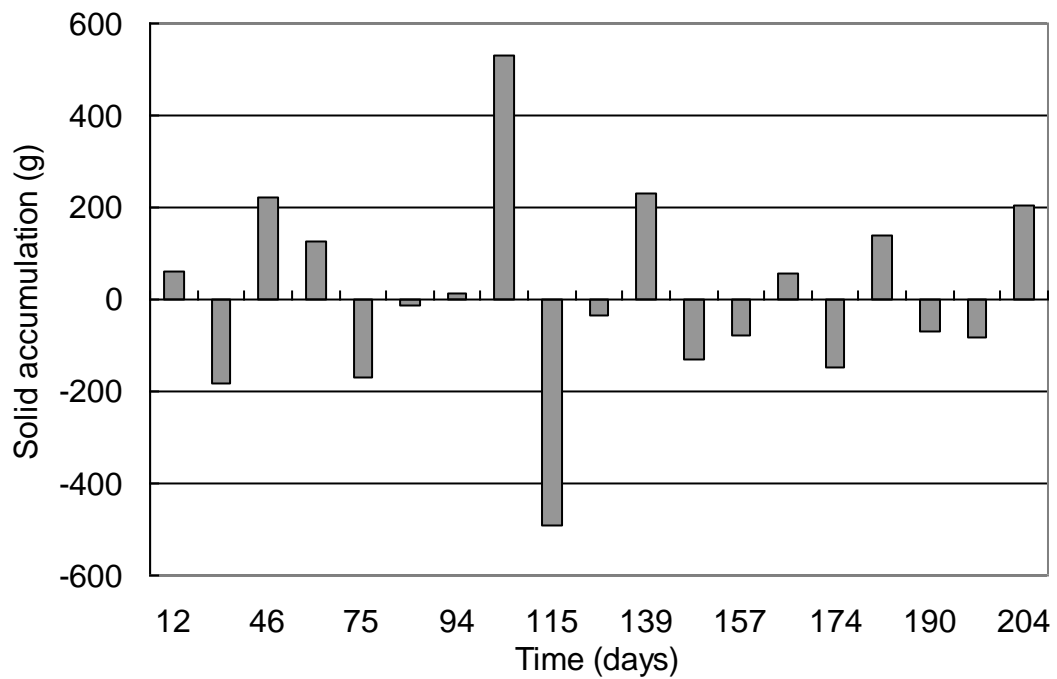
Figure 5-10. Solids accumulation in the SGBR system
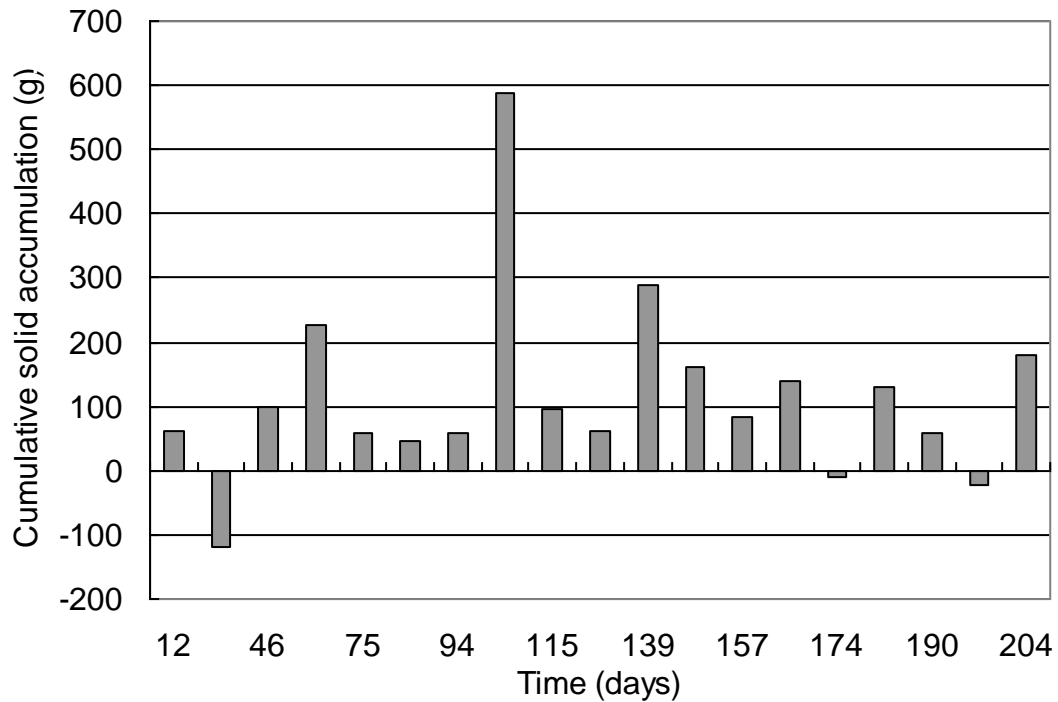
Figure 5-11. Cumulative solids accumulation in the system

Specific methane activity (SMA)

The methane activity test of granular sludge provides a valuable indication of the presence of inhibitory compounds as well as the activity state of the biomass used for the anaerobic system (Campos and Chernicharo, 1991; Fang et al., 1997; Liu et al., 2002). Several SMA tests were performed for the anaerobic granular sludge in the SGBR reactor during the operation to evaluate the activity of the granules. Moreover, the granular sludge was sampled from the two sampling ports, located in the middle and bottom of the reactor, to compare the activity of sludge at different depths.

SMA test results from the granular sludge seeding was 0.384 g COD-CH$_4$/gVSS-d which was a little higher than SMA (0.324-0.377 g COD-CH$_4$/gVSS-d) of the biomass

sampled from the operated SGBR. This was not a serious detrimental effect on the system

since all tested values were in the range of the normal SMA values from the active anaerobic

granules as shown in Table 5-6. The granular sludge sampled from the middle of the bed

depth had similar SMA values compared to the sludge from the bottom of the bed (Figure 5-

12). The differences in SMA results were often observed at different depths in the granular

bed system (Núñez and Martínez, 1999; Roth, 2003). The system operated at a high organic

loading rate (e.g., above 5 kg/m^3/d of overall average organic loading rate after the startup).

However, it reduced the differences in granule activity at different depths, due to the increase

in substrate supplied to the biomass in deeper positions (Núñez and Martínez, 1999). Figure

5-12 also shows the SMA differences between middle and bottom granules decreased as the

organic loading rate increased. Therefore, the similar SMA activities at each depth

performed in this study owed to the high organic loading rates ranging from 5.5 to 12.8

kg/m^3/d. Moreover, this similarity in activities at each depth seemed to be affected by the

periodic backwashing, providing a similar effect of mixing and moving the granular bed.

Table 5-6. Comparison of SMA results between this study and other tested biomass

Original feed	SMA (gCH_4-COD/gVSS-d)	Reference
Liquid sugar wastewater	0.9	Dolfing and Mulder (1985)
Maize starch wastewater	0.11	Dolfing and Mulder (1985)
Potato processing Wastewater	0.13	Colleran et al. (1992)
Brewery	0.08	Colleran et al. (1992)
Slaughterhouse wastewater	0.2-0.8	Núñez and Martínez (1999)
Mixture of primary and waste activated sludge	0.149 - 0.221	Vandenburgh and Ellis (2002)
Non-fat dry milk	0.083 - 0.406	Roth (2003)
Slaughterhouse wastewater	**0.324 - 0.377**	This study
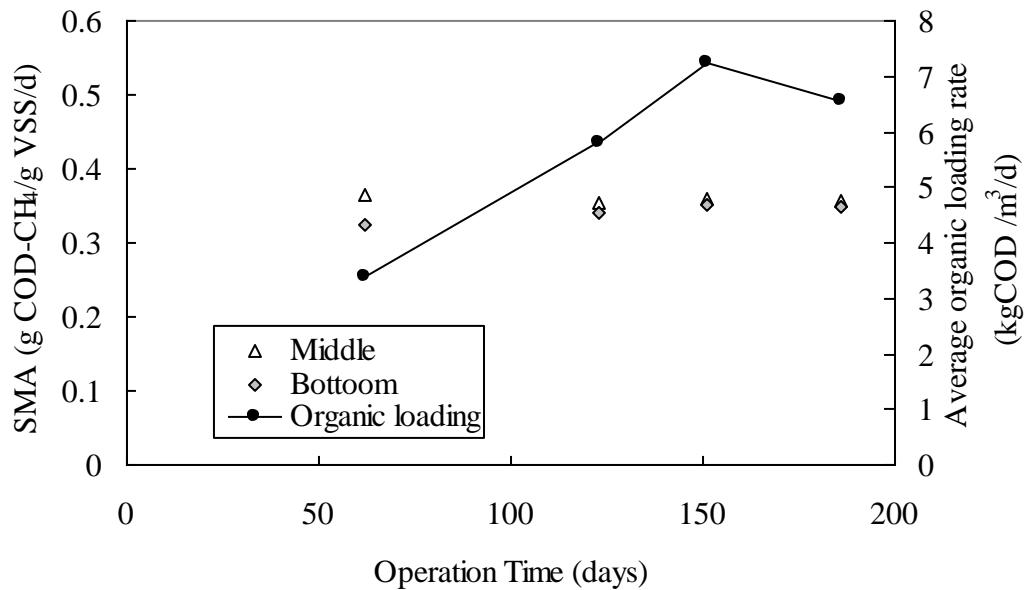
Figure 5-12. SMA of middle and bottom granules at various organic loading rates

Biomass yield calculation

The biomass yield calculations were performed, using a COD mass balance on the

SGBR system. The mass balance at steady state was determined as shown below:

$QC_{in} - QC_{out} - CH_{4\ prod} - \text{Biosynthesis} = 0$ $\qquad\qquad$ (5.6)

Where, Q: flow rate (m^3/d)

$\quad$ C_{in}: COD concentration in influent (mg/L)

$\quad$ C_{out}: COD concentration in effluent (mg/L)

$\quad$ $CH_{4\ prod}$: CH_4 production rate (m^3/d)

It was assumed that the total input COD to SGBR was the sum of the SCOD in the

discharged effluent, COD from VSS in the effluent, methane conversion of COD, and

biomass production by the consumed COD. Therefore, the above mass balance equations were expressed as shown below:

Biomass growth (g COD/d) = Q x ($TCOD_{inf}$ –$SCOD_{eff}$ – VSS_{eff} as COD)

– $CH_{4\ prod}$ as COD   (5.7)

To calculate COD equivalent of biomass, the mass of methane production and VSS discharged in the effluent were converted to COD values by the following equations:

$CH_{4\ prod}$ (g COD/d) = CH_4 production (L/d) /0.35(L/g COD)   (5.8)

VSS_{eff} (g COD/d) = VSS_{eff} (g VSS/L) x 1.42 x Q   (5.9)

The following equation expressed the biomass accumulation rate in the SGBR system, using a COD balance:

Biomass $_{accum}$ (g COD/d) = $TCOD_{in}$ (g/d) – $SCOD_{out}$ (g/d) – $CH_{4\ prod}$ (g COD/d)

 –VSS_{eff} (g COD/d)   (5.10)

Biomass yield calculations were determined by computing the accumulated biomass and dividing it by the total removed COD in the system. Considering biomass synthesis during the operation of the SGBR, VSS in the effluent is included in that calculation by assuming the complete degradation of influent solids and no accumulation of substrate in the system. Therefore, biomass yield calculation was expressed as shown below:

$$\text{Biomass yield (g VSS/g COD)} = \frac{(\text{Biomass}_{\text{accum}} + \text{VSS}_{\text{eff}}) \div 1.42 \ (\text{g VSS})}{\text{Removed COD}(\text{g})} \tag{5.11}$$

The results of biomass yield calculations during the operation are shown in Table 5-7. The average calculated biomass yield was 0.057- 0.122 at each HRT condition. The variations of the calculated yields observed at each HRT were caused by change in the organic loading rate and variations in influent characteristics. The yields from the operation over 48 hours HRTs were lower than those of longer HRT. In the same hydraulic flow rate condition, the higher yield values were calculated from the higher organic loading rate conditions. Overall biomass yields were a little lower than the reported biomass yields from the anaerobic treatment of slaughterhouse wastewater, which were 0.15-0.50 and 0.257 from UASB and EGSB system respectively (Sayed et al., 1984; Núñez and Martínez, 1999).

Table 5-7. Biomass yield at each HRT with variation of organic loading rates

Operation time (Days)	HRT (hours)	Organic loading rates (kg COD/m³/d)	Average yield per sectioned time (g VSS/ g COD $_{removed}$)	Average yield (g VSS/ g COD $_{removed}$)
51	48	2.97	0.057	0.057
62		4.35	0.058	
79		4.91	0.130	
97	36	8.23	0.077	0.122
111		6.42	0.213	
128		4.33	0.131	
136		7.71	0.103	
147	30	6.95	0.092	0.073
159		6.87	0.067	
182		6.2	0.030	
194		8.09	0.125	
203	24	5.19	0.049	0.064
210		5.44	0.031	
219		5.55	0.050	
228		6.04	0.051	
237		6.10	0.034	
244	20	4.85	0.056	0.045
251		6.43	0.043	
258		6.59	0.039	

Conclusions

The newly developed anaerobic biotechnology, the SGBR, was based on a granular

sludge system achieved effective treatment of slaughterhouse wastewater. The rapid startup

of the SGBR was shown in this study, due to 10-15 days of short duration time prior to

reaching the normal condition of the system. High organic removal efficiency (over 95% of

TSS and VSS removal) was obtained at various HRT (48, 36,30, and 24 hours), owing to the

consistent treatability of the SGBR system during the operation. This result from the SGBR

performance showed a high potential for treating slaughterhouse wastewater in less than 24

hours of HRT conditions. The stable treatment efficiency was performed at fluctuating

organic loading rates from 0.77 kg/m^3/d to 12.76 kg/m^3/d, while there was a great change in wastewater characteristics, indicating the ability of this system to satisfy variations in wastewater characteristics and maintain consistent performance. Moreover, COD removal efficiency of above 95% at these organic loading rates performed equal to or better, compared to other high-rate anaerobic system treating slaughterhouse wastewater, as shown in Figure 5-6..

The backwashing used in this study alleviated increasing hydraulic head loss periodically due to the wasting of portion of over-accumulated solids in the system. The accumulation of solids did not cause detrimental effects on the system. This is shown by a stable and high COD removal efficiency, methane production, and low VFA concentrations in the effluent. Moreover, there was no significant amount of accumulated solids, as evidenced by the solids balance calculations. Therefore, the pilot-scale SGBR operated stably, owing to effective backwashing and consistent organic removal performance. This exemplifies the good applicability of this system to slaughterhouse wastewater, and also indicates the high potential of commercialization.

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CHAPTER 6. CONCLUSIONS

This dissertation focuses on several important areas regarding opportunities for environmental protection, renewable energy, and resource recovery. All of the studies involved a start-up company, Envirotech Systems, Inc., based in Lawton, Iowa. Envirotech currently has two main products they are focusing on—tire derived rubber particles (TDRP) and the static granular bed reactor (SGBR). This dissertation documents the close association between the development and marketing of new environmental management opportunities and a research-based institution, Iowa State University.

The evaluation study for the application of TDRP to the wastewater treatment validates its feasibility for various biofilm systems proven by trickling filters, SGBR with TDRP addition and hybrid SGBR reactors. The excellent hydrogen sulfide removal (e.g., over 94% H_2S removal efficiency at 20-90 ppm H_2S in inflow during several EBRTs ranging from 20 to 67 seconds) was performed by the pilot-scale TDRP biofilter system. The comparison study between the SGBR and TDRP adding system showed the effectiveness of TDRP media compared to the anaerobic granules, due to the similar performance of four demonstrated reactors having different portions of TDRP media volume.

Results from each aspect of these studies showed a high potential of TDRP to support biological activity in a variety of treatment applications. The effectiveness of TDRP for biomass support media was proven by SEM analysis of TDRP media, as well as the performances of bioreactors. Each biotechnology using TDRP in this study could be considered a pioneer process for using the reused rubber materials, due to its successful achievements and usefulness in various aspects. It was obvious that the development of

biosystems using TDRP could not only provide a cost effective process, but also could reduce the waste management problem of scrap tires.

Results from the pilot-scale SGBR using anaerobic granules demonstrated effective biological treatment of slaughterhouse wastewater with a high organic removal efficiency. Over 95% COD and TSS removal efficiency was achieved at various organic loading rates from 0.77 kg/m^3/d to 12.76 kg/m^3/d. A consistent 95% and above organic removal was evidence to the ability of the SGBR to overcome the limitations of treating slaughterhouse wastewater, which has a high fraction of insoluble organic matter (typically 40-60%), consisting mainly of slowly degraded proteins, fats, and cellulose (Sayed et al., 1987). Effective periodic backwashing was an important element for the successful SGBR operation, since it provided the system stability with alleviation of the increase in hydraulic head loss by wasting of a portion of the accumulated solids in the system. Throughout the entire study, the consistent pilot-scale SGBR system performance at higher organic and hydraulic loading indicated a high potential of the SGBR on a full-scale facility treating slaughterhouse wastewater.

Engineering Significance

The development of biofilter systems using TDRP may be a valuable strategy for the reuse of tire rubber from scrap tires. Moreover, the application of TDRP biofilter media to the anaerobic process, whose benefits include energy savings and low sludge production, can prove profitable to treatment plants. Wastewater treatment costs could be reduced by using

TDRP for the filter media because of the low price of reused tire product compared to other commercial biofilter media products.

Hydrogen sulfide removal by the TDRP biofilter system showed exceptional achievement, due to the high removal efficiency at 20 s and greater EBRTs. The successful performance of the TDRP biofilter provides an advantage in the environmental field, resulting in a cost savings by using tire material and the effectiveness of smaller reactor volumes evidenced by over 30 g-H_2S/m^3/h of treatment capacity from this study.

The SGBR is a unique and simple downflow high rate anaerobic system using anaerobic granules. The effective backwashing strategy included controlling the backwashing intervals, the amount of backwash water, and the backwash flowrate with consideration of the wastewater characteristics and variation of wastewater flow. The achievement of effective organic removal from the SGBR at low HRTs and high organic loading rates alludes to the smaller treatment system size for the same amount of wastewater flow compared to other systems. The commercialization of the SGBR from the successful operation of the pilot-scale can provide benefits to the food processing company, owing to its simple design and operational advantages over conventional systems. The rapid startup achieved within 10 to 15 d without any specific management intervention (such as alkalinity or nutrient addition) indicates the cost effectiveness of SGBR. Other systems have required the addition of methanol for rapid startup (e.g., during the startup of an anaerobic downflow filter system treating slaughterhouse wastewater by Borja et al., 1994). Moreover, the consistent biogas production with stable and high methane composition furnishes significant energy savings (e.g. natural gas cost is about $8-12/MMbtu in the U.S.). The low biomass

yield of the pilot-scale SGBR compared to other process also provides benefits of lower

sludge disposal costs, approximately $100-170/ton dry solids (Lue-Hing et al., 1998).

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APPENDIX

Appendix A. Test results for characteristics of TDRP

Floatation and precipitation characteristics

The purpose of this preliminary test was to provide some indication of the effect of TDRP cover on downstream unit processes. For instance, if a lagoon had a TDRP cover, what would be the effect of suspended TDRP material that did not stay with the cover, but instead flowed into downstream unit process, such as an activated sludge system. The floatation and precipitation tests were performed using a 16-L cylinder reactor filled with 14-L distilled water and 700 g TDRP. This reactor was operated for 8 days with the intermittent stirring. The floating TDRP and precipitating TDRP were collected and dried to measure their respective weights for comparison.

During the period of operation, the ratio of precipitated to floating TDRP increased with time. In the beginning, the TDRP flotation film was 3 inches on the water surface. However, it was less than 0.5 inches after 7days of the operation as shown in Figure A1. During the operating period, the portion of the precipitated TDRP increased with time. Finally, the dry weight of the precipitated TDRP was 80.9 % of the total TDRP as shown in Table A1. Therefore, these results showed that the TDRP had high precipitation potential during the operation.
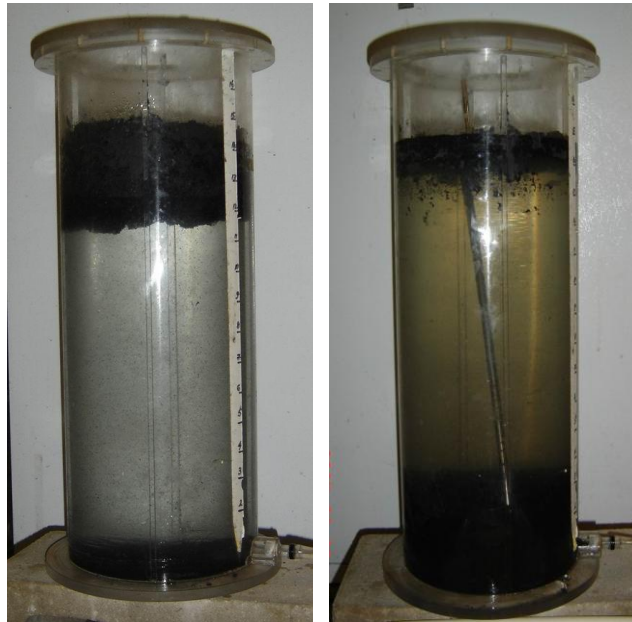
Figure A1. Differences between Day 1 and Day 8 TDRP floatation and precipitation

Table A1. Results of TDRP dry weight from the precipitation test

	Dry weight (g)	Percentage (%)
TDRP Precipitation	566	80.9
TDRP Flotation	132	19.1

SVI Test using activated sludge for TDRP addition effect

This test was performed with the activated sludge sampled from the Boone Wastewater

Treatment Plant. TDRP was added to the activated sludge in various quantities as shown in

Table 2. After dosing the TDRP, the jar was mixed with the jar test apparatus at 200 rpm for

1 min. and 120 rpm in 30 min. Afterwards, the activated sludge, mixed with TDRP, was put into the SVI test apparatus to measure the settleability.

Table A2 shows the SV and SVI results of each different TDRP dosage. There were no distinctive SV differences along with the various TDRP dosages. Therefore, the SVI results were also similar values among them. However, the COD value for each supernatant was affected by the TDRP addition as shown in Table A3. As the dosage quantity of TDRP increased, the COD value also increased. However, this increase was not too much and might be neglected, if it is applied to the high strength wastewater.

Table A2. Results of the SVI test

TDRP dosage(g)	0	27	42	58	100
SV (%)	15	13.5	14	14	15
SVI (mL/g)	74.6	64.7	69.7	69.7	74.6
Total volume (L)	4	4	4	4	4
Sludge VSS(mg/L)	2,010	2,010	2,010	2,010	2,010

Table A3. COD and TSS results of the supernatant of the jar test

TDRP dosage (g)	0	27	42	58	100
COD (mg/L)	29.4	45.0	49.4	65.1	109.4
TSS (mg/L)	8	6	7	6	6

Hydraulic and hydrodynamic properties of TDRP

A 10 L plexiglass reactor was used for the determination of hydraulic characteristics of TDRP. Figure A2 shows the schematic diagram and picture of this test. Headloss as a function of bed depth was determined in a packed column reactor receiving a variable air flow stream. The product "A" and the product "B" were tested in depth 8 to 32 inches.
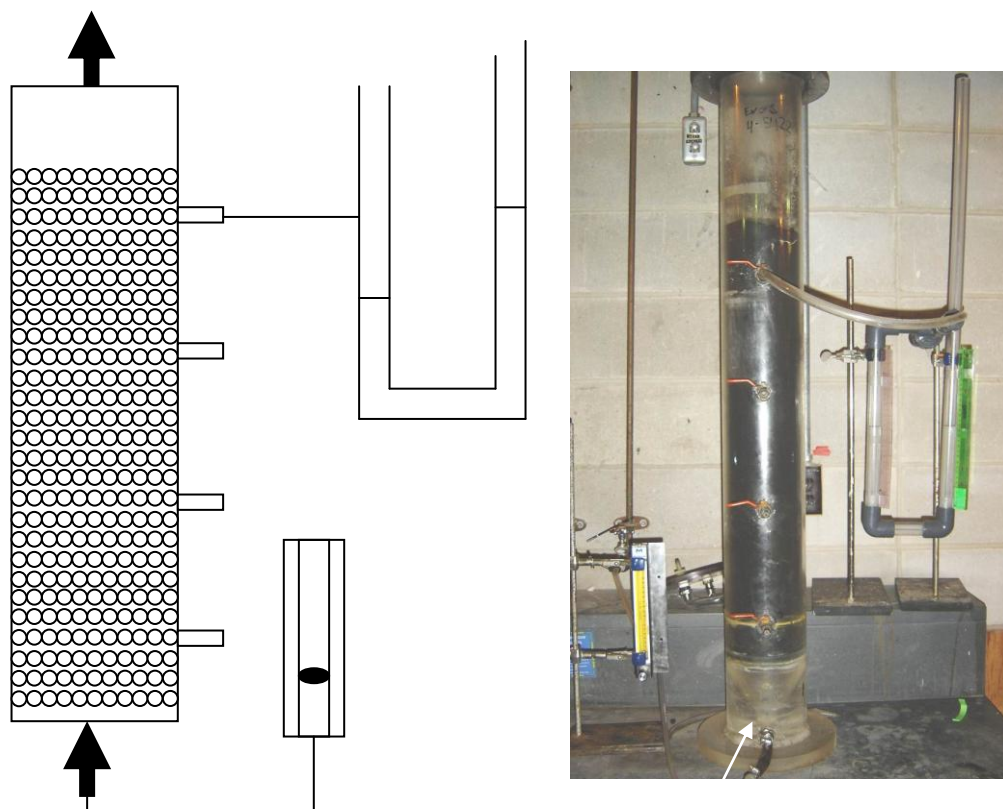
Figure A2. Configuration and picture of TDRP packed column reactor

Table A4 shows the results of the head loss as a function of bed depth. Each product showed the head loss increase with higher depth and higher air flow rate. These two products had similar head loss changes during the test. However, product "B" had a higher head loss compared to product "A" in same condition (Figure A3). Since some metal was added to product "B," it is more compact than the product "A," Therefore, product "A" had less head loss than product "B."

Table A4. Head loss results of TDRP on various depth and air flow rate   Unit: Pa

Air flow rate (SLPM)	Control	Product "A" depth (inch)				Product "B" depth (inch)			
		32	24	16	8	32	24	16	8
1	0	39.2	29.4	9.8	0	49	39.2	29.4	9.8
2	0	58.8	49.0	19.6	4.9	147	98	58.8	14.7
4	0	88.2	68.6	29.4	9.8	259.7	176.4	107.8	20.58
6	0	147.0	107.8	44.1	14.7	382.2	269.5	156.8	32.34
8	0	196.0	137.2	58.8	19.6	519.4	343	215.6	44.1
10	0	245.0	176.4	83.3	29.4	646.8	421.4	269.5	58.8
12	0	303.8	205.8	107.8	44.1	764.4	514.5	323.4	73.5

* Fluid used for nanometer: water (Density = 1.0 kg/L)
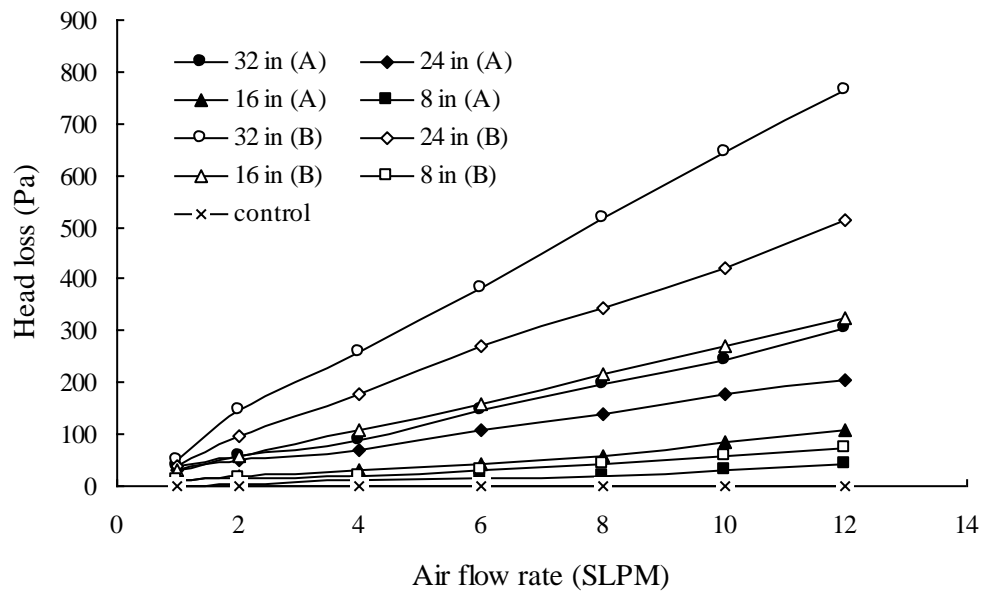
Figure A3. Head loss with various air flow rate and TDRP bed depth

Appendix B. SMA (Specific Methane Activity) Test

1. Reagents

1) Batch medium

Compound	Concentration
$NaH_2PO_4H_2O$	7.95 g/L
K_2HPO_4	6 g/L
NH_4Cl	2.8 g/L
$MgSO_4.7H_2O$	1.11 g/L
$CaCl_2.2H_2O$	0.1 g/L
Yeast extract	0.2 g/L
Trace element	10mL/L

- Trace element

Compound	Concentration	Compound	Concentration
$FeCl_2.4H_2O$	10 g/L	$AlCl_3.6H_2O$	0.09 g/L
$CoCl_2.6H_2O$	2 g/L	H_3BO_3	0.05 g/L
EDTA	1 g/L	$ZnCl_2$	0.05 g/L
$MnCl_2.4H_2O$	0.5 g/L	$(NH_4)_6Mo_7O_{24}.4H_2O$	0.05 g/L
Resazurin	0.2 g/L	$CaCl_2.2H_2O$	0.038 g/L
$NiCl_2.6H_2O$	0.142 g/L	HCl (37%)	1 mL/L
Na_2SeO_3	0.123 g/L		

2) Buffer Solution
- $NaHCO_3$: 90,000 mg/L

3) Sodium sulfide solution (0.25M)
- $Na_2S.9H_2O$: 60.04g/L

4) Deoxygenated water (Anaerobic water)
 - Make anaerobic water by flushing tap water with nitrogen gas.

5) Acetic Acid dilution
 - Dilute the acetic acid for the convenience. c.f.: 0.90 mL Acetic acid = 1g COD

2. Procedure

1) Add 10~15 mL of batch medium solution into the 250mL serum bottles

2) Add 10 mL of buffer solution

3) Add the biomass into the serum bottles : f.e. make the biomass conc. 500~1000 mg/L

4) Add anaerobic water until volume will be around 150mL (including biomass)

5) Inject the substrate (Acetic acids) with considering the F/M ratio to avoid the substrate inhibition.

6) Correct the pH to around 7.0 by adding NaOH or HCl (flushing with N_2 will further increase the pH)

7) Add 0.5 mL of 0.5 M Na_2S for reducing environment as recommended in the SMA test.

8) Flush the solution with nitrogen gas for 30 seconds when bottle is open.

9) Flush the head space in the serum bottle for several seconds just before closing it with septa.

10) Check the solution color. The solution should be white or colorless. When the solution is still pink add a little more Na_2S or flush more.

11) All the test bottles are incubated at 35^oC in an incubator shaker at 150 rpm.

12) Release the gas after 1 hour since incubating.

13) Measure the gas production and composition every several hours depending on the gas production rate.

Appendix C. Analysis results using Jump (COD removal comparison among SGBR and Hybrid SGBR reactors)

Response Column 2
Whole Model
Actual by Predicted Plot

Column 2 Predicted P<.0001
RSq=0.78 RMSE=0.1995
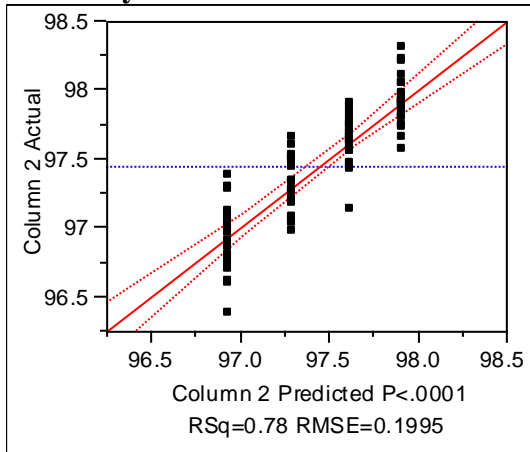
Summary of Fit

RSquare	0.779233
RSquare Adj	0.771707
Root Mean Square Error	0.199535
Mean of Response	97.44087
Observations (or Sum Wgts)	92

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	12.366687	4.12223	103.5368
Error	88	3.503643	0.03981	Prob > F
C. Total	91	15.870330		<.0001

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Column 1	3	3	12.366687	103.5368	<.0001

Residual by Predicted Plot
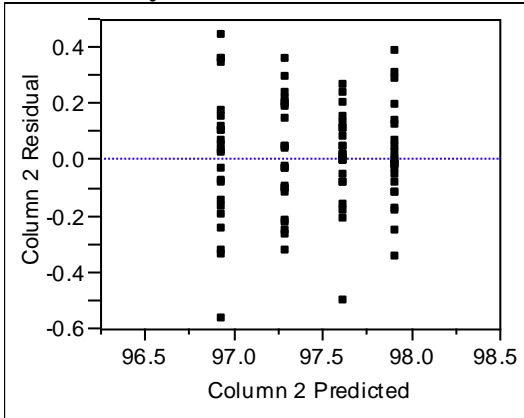
Column 1
Leverage Plot
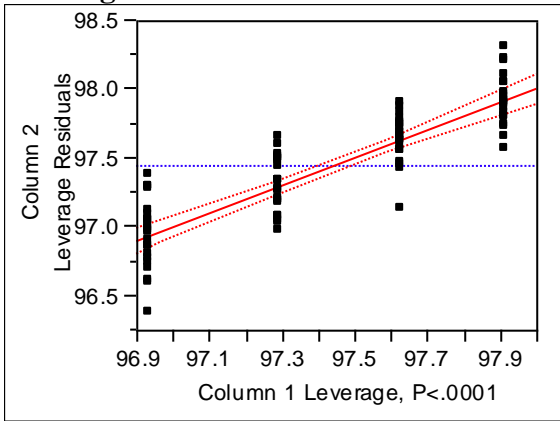
Least Squares Means Table

Level	Least Sq Mean		Std Error	Mean
R1	96.932174		0.04160588	96.9322
R2	97.291739		0.04160588	97.2917
R3	97.627826		0.04160588	97.6278
R4	97.911739		0.04160588	97.9117

LSMeans Differences Student's t

α=
0.050 t=
1.98729
LSMean[i] By LSMean[j]

Mean[i]-Mean[j] Std Err Dif Lower CL Dif Upper CL Dif	R1	R2	R3	R4
R1	0 0 0 0	-0.3596 0.05884 -0.4765 -0.2426	-0.6957 0.05884 -0.8126 -0.5787	-0.9796 0.05884 -1.0965 -0.8626
R2	0.35957 0.05884 0.24263 0.4765	0 0 0 0	-0.3361 0.05884 -0.453 -0.2192	-0.62 0.05884 -0.7369 -0.5031
R3	0.69565 0.05884 0.57872 0.81258	0.33609 0.05884 0.21916 0.45302	0 0 0 0	-0.2839 0.05884 -0.4008 -0.167
R4	0.97957 0.05884 0.86263 1.0965	0.62 0.05884 0.50307 0.73693	0.28391 0.05884 0.16698 0.40084	0 0 0 0

Level					Least Sq Mean
R4	R1				97.911739
R3		R2			97.627826
R2			R3		97.291739
R1				R4	96.932174

Levels not connected by same letter are significantly different.

LSMeans Differences Tukey HSD

α=
0.050   Q=
2.61881
LSMean[i] By LSMean[j]

Mean[i]-Mean[j] Std Err Dif Lower CL Dif Upper CL Dif	R1	R2	R3	R4
R1	0 0 0 0	-0.3596 0.05884 -0.5137 -0.2055	-0.6957 0.05884 -0.8497 -0.5416	-0.9796 0.05884 -1.1337 -0.8255
R2	0.35957 0.05884 0.20548 0.51365	0 0 0 0	-0.3361 0.05884 -0.4902 -0.182	-0.62 0.05884 -0.7741 -0.4659
R3	0.69565 0.05884 0.54156 0.84974	0.33609 0.05884 0.182 0.49018	0 0 0 0	-0.2839 0.05884 -0.438 -0.1298
R4	0.97957 0.05884 0.82548 1.13365	0.62 0.05884 0.46591 0.77409	0.28391 0.05884 0.12982 0.438	0 0 0 0

Level					Least Sq Mean
R4	R1				97.911739
R3		R2			97.627826
R2			R3		97.291739
R1				R4	96.932174

Levels not connected by same letter are significantly different.

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