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1991

Initial studies of the anaerobic sequencing batch reactor

Craig Edward Habben *Iowa State University*

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Initial studies of the anaerobic sequencing batch reactor

by

Craig Edward Habben

A Thesis Submitted to the

Graduate Faculty in Partial Fulfillment of the

Requirements for the Degree of

MASTER OF SCIENCE

Department: Civil and Construction Engineering Major: Civil Engineering (Environmental Engineering)

Signatures have been redacted for privacy Signatures have been redacted for privacy

Iowa State University Ames, Iowa

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1991

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LIST OF ABBREVIATIONS

- ASBR Anaerobic Sequencing Batch Reactor
- $BOD₅$ 5-day Biochemical Oxygen Demand
- BOD_L Ultimate Biochemical Oxygen Demand
- COD Chemical Oxygen Demand
- ERI Engineering Research Institute
- FAS Ferrous Ammonium Sulfate
- GC Gas Chromatograph
- **HRT** Hydraulic Retention Time
- mgd Million Gallons per Day
- MLSS Mixed Liquor Suspended Solids
- MLVSS Mixed Liquor Volatile Suspended Solids
- ppm Parts per Million
- rpm Revolutions per Minute
- SBR Sequencing Batch Reactor
- SCOD Soluble Chemical Oxygen Demand
- SMAR Submerged Media Anaerobic Reactor
- **SRT** Solids Retention Time
- STP Standard Pressure and Temperature
- TCOD Total Chemical Oxygen Demand
- 1KN Total Kjeldahl Nitrogen
- 10C Total Organic Carbon
- TSS Total Suspended Solids
- UASB Upflow Anaerobic Sludge Blanket

VSS Volatile Suspended Solids

WPCP Water Pollution Control Plant

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I. INTRODUCTION

The use of anaerobic processes for the stabilization of industrial wastes is seeing increased popularity. Several countries, including the United States, are showing increased efforts in the areas of research and development on different anaerobic processes. When an anaerobic treatment process is designed, implemented and managed properly, the process can offer several advantages. McCarty [24] presented some of these advantages, including:

- A high degree of waste stabilization is possible.
- Low production of waste biological sludge.
- Low nutrient requirements.
- No oxygen requirements.

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• Methane is a useful end product.

There are several anaerobic processes that have been used for industrial waste stabilization. Some of these are: anaerobic contact process, fluidized-bed anaerobic process, anaerobic filters, hybrid anaerobic processes, and upflow anaerobic sludge blanket reactor.

A key factor in the successful operation of any of these systems, and what most of them are based on, is the achievement of a solids retention time (SRT) that is much longer than the hydraulic retention time (HRT). In a 1970 paper [11], Dague stated that the minimum design SRT in an anaerobic reactor is 10 days, at a temperature of 350 C. Thus, if a system is operating at a HRT less than 10 days, solids must be retained in the digester in order to accomplish SRTs which are

significantly higher than the minimum 10 day value. In the filter processes, solid media are used to aid in retaining biomass. The anaerobic contact process uses degasification and external settling WIth solids recycle to achieve the necessary SRT.

The Anaerobic Sequencing Batch Reactor (ASBR) accomplishes solids retention without the aid of media, vacuum degasification, external clarifiers, extensive amounts of chemicals, or other mechanical systems. In essence, the ASBR is an "anaerobic activated sludge" process with internal settling and supernatant (effluent) wasting. Thus, if the ASBR can be successfully operated, it would provide a simpler process, because no external clarifier and degasification is needed as In the anaerobic contact process, and the ASBR would eliminate the expensive media required in submerged media anaerobic reactors

II. OBJECTIVES AND SCOPE OF STUDY

The ASBR is a new technology for treating wastewater. Thus, the first priority of the research was to develop a reactor design that permits the feeding, mixing and reacting, settling, and wasting required for efficient operation of the process under anaerobic conditions.

The design and operation of the reactor for this research was the only system known of its kind (however, since the beginning of this research, two other systems labeled as anaerobic sequencing batch reactors have surfaced in the literature [18,19]). Therefore, initial operation was required to determine if It was possible to maintain a settleable microbial mass which can achieve solids retention times greater than 10 days at 350 C at non-trivial organic loadmgs.

Once it was determined that sufficient solids retention times could be achieved, the focus of the remaining research was centered on developing a feel for the essential fundamentals associated with running an ASBR. This was accomplished by studying the effects of various organic loading rates as well as different hydraulic loading rates. Also, the response of the reactor to different sequence scenarios and other seen or unforeseen factors associated with the operation of the reactor was helpful in determining the fundamentals of the system.

The following were the specific objectives of the research:

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1) Design a reactor to perform the sequencing operations under anaerobic conditions while allowing for accessibility to

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perform the needed tests to monitor and control the reactor's operation.

- 2) Determine if a settleable, non-granular microbial mass can be internally maintained in the reactor, while achieving the required minimum solids retention time of 10 days at a temperature of 350 c.
- 3) Develop and maintain a microbial mass without chemically stimulating granulation (however, if granulation occurs so much the better, in terms of settling capabilities), flocculation or coagulation.
- 4) Determine the maximum organic loading rates that can be efficiently removed by the system.
- 5) Determine the performance of the system at different HRTs.
- 6) Determine the fundamentals associated with successful operation of the system.
- 7) Develop an example design to treat a wastewater stream by applying an ASBR.
- 8) Develop and stimulate ideas for further research.

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The primary purpose for the development of this new process was to create a system that can be easily maintained without significant added operations. Therefore, the simplest scenario of operation was researched. In other words, the system was operated without attempting to develop granular sludge, or chemical enhancement by coagulation and flocculation. The easier a system is to

operate, the more the person in charge of its operation will like it and want to use it.

III. LITERATURE REVIEW

The literature review will focus on processes, concepts and history directly associated with the development and operation of the ASBR. General anaerobic treatment concepts and fundamentals will not be discussed but referenced [6,8,24,25,26,27,30,35].

A. Early Developments of Anaerobic Treatment

The application of anaerobic treatment processes to industrial wastes has been an uphill battle. In the 1910s anaerobic treatment was first applied to separate sludge digestion [20]. This led to a great deal of development and research in separate sludge digestion during the 1920s and 1930s [21,31,41], which was good for stimulating greater interest in determining some of the fundamentals, but lead to a philosophy that anaerobic treatment was only suitable for sludge digestion. Bach stated firmly that anaerobic bacteria should only be adopted for sludge treatment and not for treating the liquid portion of the sewage [5].

Thus, much of the anaerobic research over the next twenty years was based on sludge digestion. The research concerned things such as temperature effects, kinetics, gas production, methane bacteria, detention time and loading, and volatile acids limits [8]. This work was very important for understanding the fundamentals of anaerobic

treatment, but at this point anaerobic processes were not generally applied to raw waste streams.

In the meantime aerobic treatment, primarily activated sludge and modifications thereof, developed as the principal means of treating the raw waste stream of concern at the time, low strength, relatively dilute domestic sewage [5]. Anaerobic treatment was limited to certain high strength BOD wastes, particularly wastes from fermentation industries [40] and, as mentioned before, sewage sludge.

In 1950 W.J. Fullen, encouraged by the results of Buswell [1] on treatment of yeast and gram industrial wastes [32], used the ideas of activated sludge (mixing an incoming waste stream with an already biologically "active" sludge), and applied them anaerobically to the treatment of slaughter house wastes. This development, latter labeled the "Anaerobic Contact Process," was very instrumental in stimulating the concepts of anaerobic treatment for the raw industrial waste streams because the concept of solids separation provided for long solids detentions and much shorter liquid detentions.

B. Anaerobic Contact Process

The soon to be called "anaerobic contact process" was first applied to packing plant wastes in Austin, Minnesota in a study by Geo. A. Hormel Co., as reported by Fullen in 1953 [15]. The continuous system involved introducing the incoming waste with an already active anaerobic sludge, allowing for contact time, settling the solids out of the

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mixed liquor effluent stream, and returning the settled sludge to the main reactor.

BOD removals were reported by Fullen to be 95 to 96% with BOD loadings ranging from 0.067 to 0.084 lb per cu ft per day (1.07 to 1.35 $g/L/day$). Detention times were 24 hours or less and volatile solids loadings were shown to be 0.061 to 0.081 lb per cu ft per day (0.98 to 1.30 $g/L/day$.

In 1955, Schroepfer *et al* [33] reported on further anaerobic contact studies at the Geo. A. Hormel, Austin, Minnesota, site. One of the main concerns of these researchers, from the initial studies, was the efficiency of solids separation. Therefore, many of the investigations were designed to improve solids separation. These investigations included adding fly ash to the mixed liquor as a weighting agent, implementing a vacuum evacuation process followed by gravity settling, and installing a sludge flotation system. The fly ash seemed to help but was reported as impractical. Flotation worked but was not as efficient or economical as the vacuum evacuation process. The evacuation process followed by gravity settling showed the best results by producing solids removals up to 99.7% . Figure 1 shows a schematic of the anaerobic contact process as applied at the Austin site. The following is an abbreviated version of the summary presented by Schroepfer *et al. [33]:*

• A process has been developed which for this type of waste is capable of accomplishing removals in 5-day BOD of 95 per cent

Figure 1. Schematic flow diagram of anaerobic contact process as **applied to packinghouse waste at the Geo. A. Hormel & Co. plant in Austin, Minnesota [19]**

and in suspended solids of 90 per cent at loadings up to 0.20 lb of BOD per cubic foot of digester volume per day (3.21 g/L/day) .

- These removals can be accomplished with detention periods in the digester of less than 12 hr. based on the raw flow...
- With equal or greater removals the process can be loaded up to four times the rate possible for conventional aerobic processes.
- Contact between the raw waste and the biologically active sludge, measured both in time and surface area of the sludge particles, appears to be an essential of the process. The new method has been denoted as an anaerobic contact process. Reference to the method as the anaerobic activated sludge process appears to be descriptive of the actions involved.
- The efficiency of the process was markedly reduced when the degree of mixing in the digester was reduced to 20 turnovers per day (50% reduction in turnovers)(p. 485).

Other interesting observations reported by Schroepfer *et al.* include [33]:

- An optimum digester solids concentration ranged from 10,000 to 11,000 mg/L total volatile solids and $13,000$ to $14,000$ mg/L total suspended solids.
- Solids concentrations above 15,000 mg/L resulted in problems of hindered settling.
- The settled sludge pumped from the settler was low in solids content requiring large volumes to be pumped in order to maintain digester solids concentration.

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• Construction costs can be as much as 50% of those costs associated with an aerobic plant but operation and maintenance costs are slightly higher than the aerobic trickling filters.

Also in 1955, Steffen [40] reported on the anaerobic contact process applied to meat packing wastes at the Wilson $\&$ Co. plant in Albert Lea, Minnesota. Steffen noted that gases entrained in the solids caused extreme variations in the solids settling, depending on the loading of the process, volatile acids concentration, solids concentration, and other factors. This problem led to what Steffen reported as the "first waste treatment plant incorporating de gasifiers in the treatment process." A 20-in (Hg) vacuum was applied on a baffled cascade arrangement in an elevated degasifier. The liquor pulled from the digester splashes down on a series of slats to aid in the release of the entrained gases. A schematic of the Albert Lea process is shown in Figure 2.

Some of the results reported by Steffen include [40]:

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- BOD reductions of the typical packing plant wastes can be reduced by 95% and suspended solids can be reduced by 97%. This is at a HRT of 12 hours, a loading rate of 0.22 lb of BOD per cu ft per day (3.53 g/L/day) , and a temperature of 95° F.
- The anaerobic contact process can be built for two-thirds the cost of a conventional two-stage trickling filter plant. However, operating costs are slightly higher than the tncking filter set-up due to the power requirements for degasification and mixing.

Figure 2. Profile of the meat processing waste treatment plant for Wilson & Co., Inc., at Albert Lea, Minnesota [21]

- Due to exceptionally light sludge, conventional sludge scrapers could not be used to remove sludge from the separators, instead suction-type devices were used to remove the high concentrated sludge.
- Sludge return rates vary from 1 to 3 normally but can range up to 10 times this during low flows in order to maintain the solids in the digester.
- High solids concentration is an advantage, but can present sedimentation problems when in the 1.1 to 1.6% suspended solids range.

Coulter *et al.* reported in 1957 on a modified version of the anaerobic contact process [3]. This system designed to treat sewage involved an unmixed contact chamber followed by an upflow rock column. The raw waste was pumped into the bottom of the contact chamber where it is brought into contact with biologically active sludge at which time coagulation and bio-precipitation take place. The flow is then upward through the sludge blanket. The column was used to further aid In solids separation and BOD removal.

Coulter *et al.* made the following conclusions and comments [3].

- Anaerobic sludge is extremely fragile and unless mechanical stirring is carefully regulated the particles are shattered and form a fine suspension difficult to separate.
- An inoffensive effluent low in BOD and very low in suspended solids was produced in the laboratory with a simple anaerobic contact system.

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• From the data thus far obtained it is difficult to make a case for the rock column. Some additional BOD and suspended solids removals are obtained, and the rock was especially beneficial during the period of upset... $(pp. 476-477)$.

In 1959, Schroepfer and Ziemke reported on a research program that studied the factors affecting the efficiency and economics of the anaerobic contact process as applied to various sewages and industrial wastes [32]. They stated that effective operation depends on two different actions: "(a) the adsorption and subsequent stabilization of organic matter, by an active biological culture, which takes place In the contact portion of the process and (b) the effective separation and concentration of the active sludge particles from the treated liquor which takes place in the separation portion of the process." Other results reported by Schroepfer and Ziemke include [32]:

- Good settling separation could possibly be achieved without degasification at low contact unit loadings.
- For a 0.20 lb of BOD per cu ft per day (3.21 g/L/day) loading rate on packing plant, synthetic milk, and fatty acid wastes, a removal rate of 90% could be achieved. Wood fiber wastes reductions were considerably lower.
- Sludge age has a consistent effect on the BOD removal. A decrease in sludge age IS followed by a decrease in removal rates.
- The maximum solids concentration obtainable is dependant on the concentrations achieved in the separator. There is some

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limiting concentration of suspended solids that can be used efficiently and appears to be in the 10,000 to 15,000 ppm range.

• Efficient mixing is necessary to achieve good contact, but violent mixing tends to break up the fine particles. The flocs, however, reform after mixing is stopped.

In 1960 and 1961, Steffen and Bedker reported on results of the first full-scale anaerobic contact process [38,39]. The anaerobic contact process, as previously described, was supplemented with oxidation ponds to polish the effluent. The anaerobic portion was able to achieve average BOD removals of 90.8% at an average loading rate of 0.156 lb of BOD per cu ft per day (2.50 g/L/day) with an overall removal of 98.2%. This was accomplished at a HRT of 12 to 13 hours and a digester suspended solids concentration ranging from 7,000 to 14,000 mg/L. One of the noted problems of the operation was the depth of the sludge blanket in the separator. If the sludge was highly active, the depth of the sludge blanket would increase to the point of over flowing the effluent weirs. Thus, the operator was required to implement evasive action to prevent excessive solids loss.

Dietz and Clinebell presented a paper in 1965 [13] on design considerations for anaerobic contact systems. They suggested maintaining suspended solids in the digester in the range of 8,000 to 12,000 mg/L and applying organic loads in the range of 0.12 to 0.20 lb of BOD per cu ft per day $(1.92 \text{ to } 3.21 \text{ g/L/day})$ with a HRT of 15 hours. They also stated that up to 1/3 of the produced gas is removed in the degasifier.

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Dietz and Clinebell were one of the first to report on problems due to corrosion. They stated that due to the corrosive nature of the gas that the degasifier and associated apparatus, such as the splash trays, vacuum pump, connecting piping, orifice meters, etc., be coated with "Heresite," galvanized, or made of stainless steel. The special materials involved in the corrosion prevention were described as more expensive. They also alluded to operational problems concerning rising sludge in the separator and maintaining temperatures in the recycled sludge.

In 1980, van den Berg and Lentz reported that performance and stability of the anaerobic contact process depended markedly on waste composition, on whether or not inoculation was used (inoculation consisted of added municipal sewage digester liquid in the amount of 10% of the feed rate), and indirectly on solids retention time [42]. Maximum loading rates were found on complex wastes of rum stillage and simulated sewage sludge to be 0.54 to 0.62 lb of COD per cu ft per day $(8.7 \text{ to } 10 \text{ g/L/day})$. Non-nutrient enriched, carbohydrate-rich wastes could only be loaded at 0.12-0.19 lb of COD per cu ft per day (2- 3 g/L/day). However, some nutrient deficient wastes were treated more successfully when higher solids retention times were maintained.

Shin *et al.* reported on the anaerobic contact process as applied to bakery wastes [34]. They indicated that a long SRT is the most important parameter for a stable and efficient contact system. They showed BOD reductions of 97.9% and suspended solids reductions of 94.3% at a loading rate of 0.190 lb of COD per cu ft per day (3.05

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 $g/L/day$, a SRT of 50 days, but a high hydraulic retention time of 7.8 days.

Shin *et al.* also alluded to some of the disadvantages of the anaerobic contact process. One being the difficulties encountered in operating the clarifier because of mechanical problems with the sludgereturn equipment. Another disadvantage being the overall general process complexity.

c. ASBR Development

The fundamental knowledge of the anaerobic contact process described above is very important for the development of the ASBR. Principles such as biological contact, solids retention, mixing, loading rates, and settling characteristics are directly applicable to the ASBR. However, the ASBR simplifies the process by removing the external systems of degasification and clarification. In order for this to be feasible, factors need to be determined and implemented that allow for internal settling to take place without the aid of degasification.

In 1966, Dague *et al* [10] were the first to report on the successful operation of a system with internal clarification in the absence of degasification. Dague's research system involved a single reactor in which feeding, mixing and reacting, settling, and decanting occurred [7]. Dague *et al* stated that "this research definitely demonstrated that biological flocculation and efficient solids separation

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by gravity settling can be achieved in anaerobic systems [11]." Other conclusions reported included [11]:

- The degree of mixing had a significant effect on the anaerobic flocculation.
- Settling problems due to gasification were not a problem in the research when bio-flocculation was occurring.
- Anaerobic activated sludge can be developed with similar flocculant characteristics as aerobic activated sludge

Also in 1966, Dague *et al.* reported that a readily settleable sludge was occurring when the metabolism of the batch fed substrate was complete and the microorganisms were in their endogenous phase [10]. At this point little gasification would be occurring and thus, there would be fewer attached gas bubbles to float the flocs. This would explain why the early anaerobic contact process had troubles with solids separation. When the mixed liquor was entering the solids separator it was still very active and producing gas. Therefore, the solids were floating to the surface because of the large amounts of attached gas bubbles. The degasification was able to help separation because it stripped the gas bubbles away as soon as they developed.

From Dague's research and others it appears that biological flocculation is important for good solids separation and clarification. McKinney, in 1958, stated that even though much of the research had been conducted on activated sludge, in terms of flocculation characteristics, the same flocculation processes also occur for anaerobic

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systems [28]. McKinney reported several of these important flocculation principles as related to the process system, including [28]:

- Energy content of the system
- Mass of inactive microorganisms
- Mass of active microorganisms
- Salt concentration
- Inorganic and organic colloids
- Agitation
- pH

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D. Applicable Anaerobic Treatment Technology

The anaerobic treatment process can be very complex in terms of the numbers of different types of microorganisms required for a specific waste treatment [23]. A diverse and closely dependant group of bacteria needs to be developed and mamtained to bring about complete conversion of substances to methane gas. In order to maintain this consortium a SRT needs to be achieved that is at least as long as the regeneration time of the slowest growing microorganism. At steady state conditions the SRT is defined at follows [14]:

SRT = Total Biomass in the system Biomass daily wasting rate

Dague's work helped establish a minimum effective solids retention time of 10 days when operating at a temperature of 35° C [7]. Parkin [30] recently stated that "SRT is now recognized as the most important parameter for system design and operation because it more accurately defines the relationship between the bacterial system and digester operating conditions..." This statement was made concerning digesters, but the same holds true for any anaerobic process. Parkin also reports on systems with higher SRTs being able to handle toxins and other modes of upset better than the systems with low SRTs.

Temperature also plays an important role in process operation. "Temperature affects the regeneration rate of the microorganisms. As temperatures decrease, regeneration times increase. Thus, temperature affects the minimum and critical solids retention times required in anaerobic systems [2]." Therefore, when operating at lower SRTs, temperature is very critical, but, as reported by Dague *et al ,* organic removal rates are independent of temperature at SRTs higher than the regeneration time of the slowest growing microbial species [11].

Mixing is generally considered necessary in suspended growth biological systems, especially contact processes like the anaerobic contact process and the ASBR. Dague stated that stabilization cannot occur unless the bacteria are brought into contact with the food, and the most effective way to do this is by mixing [6]. The amount and type of mixing, though, is somewhat up for debate.

The most popular type of mixing in anaerobic systems seems to be mechanical by means of turban or propellor-type systems. However, as mentioned early, mixing of this type that is too violent can

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break up the fragile biological flocs [32], which are important in setthng systems. In 1954, Morgan reported on an accelerated digestion process using recirculation of diffused gas throughout the digester [29]. The recirculation successfully brought the raw sludge and seed sludge into intimate contact.

Generally, it is thought that the more mixing the better. However, Dague *et al.* reported improved biological flocculation and solids separation from the initiation of intermittent mixing [11].

The development and use of granular sludge appears to have significant advantages over flocculant-type sludge. The two majors advantages are, 1) the ability to stay intact under fairly extreme cases of hydraulic stress, and 2) the ability to settling more readily because of higher specific gravity [16].

Non-granular material, such as waste activated sludge, anaerobic digested sludge, and cow manure, has been successfully used to provide inoculum for developing and cultivating granular sludges [17,43,44]. However, in some instances the granular sludge was found to dis aggregate when treating a waste other than that used as a inoculum [4].

Hulshoff Pol *et al.* reported several factors that affect the granulation process in anaerobic treatment, includmg [17]:

• Environmental conditions such as nutrient availability, temperature, pH, and the type of wastewater.

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• The type of seed sludge with respect to it specific activity, its settleability, and inert fraction.

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• Process conditions applied during start-up such as loading rate, amount of seed used, and extent of overloading.

E. Other ASBR Research

As mentioned before, two systems labeled as ASBR have been cited in the literature since this research was started. In 1989, Ketchum *et al.* reported on an application of an ASBR for treatment of coal conversion wastewaters for the Department of Energy [19]. The report showed favorable results on a phenol waste stream but not on a synthetic coal conversion waste stream. The anaerobic system was operated based on previous work done with aerobic sequencing batch reactors (SBR). The reactors generally utilized a granulated sludge. The main type parameters studied were time lengths of fill and react periods. COD loading rates and reductions were not necessarily given on the 4 and 2 liter reactors that were operated. Also, the HRT used was quite often significantly higher than the minimum SRT of 10 days as described earlier.

Kennedy *et al.* reported on an anaerobic sludge blanket sequencing batch reactor [18]. They also, in certain ways, based their anaerobic operation on studies of aerobic SBRs. The system utilized a granular sludge and supernatant recycle during the fill period. Otherwise no mixing was performed on the reactor contents. Therefore, the system described by Kennedy *et al.* is basically an anaerobic sludge blanket process, not an ASBR.

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IV. EXPERIMENTAL STUDY

A. Experimental Set-up

1. ASBR design, mechanics, and equipment

A single reactor was designed by the author based on the ideas of Dr. Richard Dague and with consultation from William Harris (coworker/research assistant at Iowa State University). The reactor was constructed by the ERI Machine Shop located on the Iowa State University Campus. A second identical reactor was constructed after initial successful operation of the first system.

The main reactor body, shown in Figures 3 and 4, IS made of plexiglas and had an effective volume of 14 liters (0.49 cu ft). The reactor is 91.44 cm tall (36 in), and has an inside diameter of 13.97 cm (5.5 in) and a wall thickness of 0.64 cm (0.25 in). A top flange, 22.86 cm (9 in) in diameter and 1.27 cm (0.5 in) thick, is attached by 12- 0.952 cm x 3.81 cm $(3/8 \text{ in } x \ 1 \ 1/2 \text{ in})$ hex-head bolts to a similar flange that is glued to the reactor. The flanges are sealed by a 0.318 cm (0.125 in) O-ring which fits into a groove in the reactor flange. A bottom flange is similarly attached and sealed at the bottom of the reactor.

Nine evenly spaced ports are located on the front of the reactor. The ports consist of plexiglas tubing and are 0.159 cm (0.0625 in) thick. The ports are glued into holes in the side of the main reactor and are reinforced by 2.54 cm x 2.54 cm x 0.635 cm $(1 \text{ in } x \text{ } 1 \text{ in } x \text{ } 0.25 \text{ in})$ plexiglas plates. These ports are used for feeding, sampling, and

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Figure 3. Section views of the ASBR

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Figure 4. DImensional view of the ASBR

wasting. A 1.27 cm (0.5 in) glass manometer tube, located on the side of the reactor, is interconnected with these side ports to aid in accurately detecting the liquid level inside the reactor (the view of the reactor contents can be obscured due to the collection of sludge and sulfur on the inside wall).

Two ports in the top flange of the reactor provide for movement of biogas, foam, and liquid, in and out of the reactor, as will be described later in this section. Another port located in the bottom flange provides for solids wasting and draining of the reactor. These ports are also made of 1.27 cm (0.5 in) plexiglas tubing and glued mto the respective flanges. The position of the main reactor and the following components which made up a complete system, can be seen m Figure 5.

The main component used to mix the reactor contents is a stone diffuser¹ which rests on the bottom flange of the reactor. The diffuser is fed by a 1.27 cm (0.5 in) stainless steel pipe, which is coupled at the top of the reactor by a compression fitting. The compression fitting is threaded into the top flange of the reactor and sealed with teflon tape. The gas recycle pump², fitted with a size 18 pump head³, is connected to the stainless steel pipe by tygon tubing. The gas recycle pump draws biogas from a foam separation bottle4 , which draws biogas from

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¹Penn Plax 3" Disk Bubble Diffuser.

²Masterflex peristaltic pump (6 to 600 rpm), Cat No. L-07553-20, Cole-Parmer Instrument Company, Chicago, Illinois 60648.

³Masterflex pump head (size 18), Cat No. L-07018-21, Cole-Parmer Instrument Company, Chicago, Illinois 60648.

⁴ Aspirator bottle (outlet/tubing), Cat. No. 02-972F, Fisher Scientific Company.

Figure 5. Schematic diagram of the overall setup for two complete systems

the ports in the top of the reactor. The four liter foam separation bottle is used to hold the occasionally developed foam until it returns to a liquid state. Once the foam collapses the liquid flows back into one of the top ports of the reactor. The necessity of the foam separation bottle is to prevent any solids that may be carned in the foam from plugging the inside of the stone diffuser. The operation principles of the foam separation bottle are shown in Figure 6.

The batching process of the system under anaerobic conditions adds an extra twist to the mechanics of the process by requiring the presence of displacement columns⁵. The main function of the displacement columns is to provide for the displacement of biogas as the reactor is filled and emptied at separate times. As the reactor is emptied, biogas is pulled from the first displacement column, raising the liquid level m this column. Simultaneously liquid moves from the second column to the first column. When the reactor is fed, the opposite occurs. Gas is pushed out of the reactor into the displacement columns and so on. Thus, the active volume in each displacement column must be at least as great as the largest volume that is wasted from the reactor at any one time. The configuration and operational aspects of the displacement columns are shown in Figures 7 and 8, respectively. The original displacement columns were smaller and did not provide enough displacement volume. The larger columns, shown in the figures, provides enough displacement volume to displace the entire reactor.

⁵Manufactured by ERI Machine Shop, Iowa State University, Ames, Iowa 50011.

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Figure 6. Operational steps of the foam separation bottle

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Step 1: Milk feed is fed into reactor displacing biogas out of the reactor head space and into the first displacement column via the foam separation bottle. Thus liquid is transferred from column one to column two.

Step 2: Produced and displaced biogas have pushed the liquid level in column one down to the angled portion of the exit tube and thus, blogas is pushed to the gas meter.

Step 3: As supernatent is wasted out of the reactor, biogas is pulled from column one to displace the supernatant. Thus, the liquid level in column one rises and the liquid level in column two falls.

Figure 7. Operational steps of the displacement columns

As biogas is produced throughout the operation of the reactor the effect is the lowering of the liquid level in the first displacement column. When the liquid level in displacement column one moves below the angled exit tube, biogas is released from the column and moves through the air lock, hydrogen sulfide gas scrubber, gas sampler, and finally through the gas meter where the biogas production is measured.

The air lock consists of a 0.95 liter (32 ounce) wide mouth glass bottle partially filled with water and sealed with a rubber stopper. Two, 0.952 cm (0.375 in) glass tubes are inserted into the stopper, one is connected to tubing from the displacement column and penetrates into the water in the bottle. The other tube just penetrates the stopper and is connected to tubing leading to the gas scrubber. Thus, biogas flows from the displacement column and through the air lock bottle, but air could not flow in the reverse direction.

The gas scrubber also consists of a 0.95 liter (32 ounce) wide mouth glass bottle. The bottle is filled with pieces of sponge soaked in ferric oxide. Again, two 0.952 cm (0.375 in) glass tubes are inserted into a rubber stopper, the influent tube extends to near the bottom of the scrubber and the exit tube just penetrates the stopper.

The 50 ml gas sampler⁶ is fitted with a septum to facilitate biogas sampling. A Wet-Test Gas Meter⁷ positioned after the gas sampler is used to measure the biogas. The biogas exiting the gas meter is drawn

⁶Manufactured by the Iowa State University Glass Blowing Shop, Ames, Iowa 50011.

⁷Precision Scientific Inc, Cat. No. 63115, Chicago, Illinois 60647.

through a piping system by an exhaust fan. The relative locations of the air lock, gas scrubber, gas sampler, and gas meter are shown in Figure 9.

The feeding of the influent stream is accomplished using a precise ten-turn potentiometer, peristaltic pump8, fitted with a size 16 pump head⁹. The wasting peristaltic pump¹⁰ has a less precise, single turn potentiometer and utilizes a size 18 pump head.

Two microprocessor-based timer/controllers¹¹ are used to turn pumps on and off at the required intervals. One timer is used to control the gas recycle pumps and the wasting pumps for both systems, and the other is used to control the feed pumps for each system.

Tygon tubing is used to connect the various components of the system. The 0.952 cm x 0.159 cm $(0.375$ in x 0.0625 in) sized tubing is fitted around ports and glass tubing with an 1.27 cm (0.5 in) outside diameter, 0.952 cm x 0.159 cm $(0.375$ in x 0.0625 in) tubing is fitted to the glass tubing on the air lock and gas scrubber, and 0.476 cm x 0.159 cm (0.1875 in x 0.0625 in) tubing is fitted to the gas sampler. To close off the tygon tubing fitted on the side ports of the reactor, the tubing IS doubled over and clamped with Hoffman screw clamps¹². Tygon tubing is also used for pump head tubing. The 0.794 cm x 0.159 cm (0.3125 in

10Masterflex penstaltic pump (1 to 100 rpm), Cat. No. L-07553-30, Cole-Parmer Company, Chicago, Illinois 60648

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⁸Masterflex penstaltic pump (6 to 600 rpm), Cat. No. L-07520-25, Cole-Parmer Instrument Company, Chicago, Illinois 60648.

⁹Masterflex pump head (size 16), Cat. No. L-0716-21, Cole-Parmer Company, Chicago, Illinois 60648.

¹¹ Cat. No. L-08614-00, Cole-Parmer Company, Chicago, Illinois 60648 ¹²Cat. No. 05-875-A, Fisher Scientific Company

Figure 9. Schematic diagram of the air lock, hydrogen sulfide gas scrubber, gas sampler, and gas meter

x 0.0625 in) and 0.318 cm x 0.159 cm (0.125 in x 0.0625 in) size tubing fits the size 18 and 16 pump heads, respectively. Interchangeable polyethylene connectors 13 are used to connect the different sized tubing and also the pump tubing.

A refrigerator, located just outside the 350 C room in which the reactors are operated, stores the milk feed. The bottom portion of Figure 5 shows the refrigerator and feed pumps. In the top part of Figure 5, which includes the reactors of the two systems, the portion of the setup contained in the 350 C room is shown. In order to prevent the milk in the substrate from spoiling as it is pumped across the floor of the hot room, the feed tubing is incased by an insulated cold water jacket. Cold water is pumped by a peristaltic pump¹⁴, with a size 18 pump head, out of the freezer portion of the refrigerator, through the cold water jacket and back into the freezer where it is recooled.

The lab scale version of the ASBR is much more complicated than a scaled up application would be. In an actual application the reactor might have a plastic cover which would pull down upon wastmg of supernatant or mixed hquor and balloon up as the reactor is filled and/or as gas is produced. Or the reactor might have a floating cover which would move up and down freely to accomplish the same task. This would eliminate the displacement columns and foam separation apparatus. These two things alone are responsible for many of the complications of the system in terms of connections and air leaks.

¹⁴Masterflex peristaltic pump (50 to 600 rpm), Cat. No. L-07553-50, Cole-Parmer Instrument Company, Chicago, Illinois 60648

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¹³Cat. Nos 15-315-A, 15-315-B, and 15-315-C, Fisher Scientific Company

2. ASBR operation

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The sequential operation of the ASBR involves four steps: feeding, mixing and reacting, settling, and supernatant wasting. This sequence could be run once a day or as many times as possible per day with the limitations being adequate settling and reacting time allowances. The sequential operation of the reactor during these steps is shown in Figures 10 and 11.

Feeding can be continuous throughout the nonsettling portion of the sequence, feeding can be done in one batch near the beginning of the sequence, or it can be accomplished by several batch feedings throughout the cycle

Mixing and reacting occurs from the time wasting is complete to the beginning of the settling period (although biodegradation reactions will actually be continuous throughout the sequence). Mixing is accomplished by pulling biogas off the head space at the top of the reactor, via the foam separation bottle, and pumping it back through the porous stone located in the bottom of the reactor. This produces a rolling effect on the contents of the reactor, and is a more gentle type mixing as compared to the mechanical type mixing. Two main types of mixing might be implemented. One is continuous mixing and the other is intermittent mixing.

Settling is the most temperamental part of the sequence. Generally if significant degradation is still occurring in the biomass, the sludge will not settle effectively because biogas being produced rises to the top, taking the biomass with it. Therefore, the settling period

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Figure 11. Sequential operation of the ASBR, Steps 3 & 4

should not begin until the feed has been significantly degraded. The minimum time required for settling is governed by the height of the effluent port and the settling velocity of the sludge/supernatant interface.

Generally, for maximum solids retention, wasting can begin once the sludge/supernatant interface is below the effluent port. However, sometimes small flocs not caught in the settling mass will still settle but at a somewhat slower rate. Therefore, it may be beneficial to wait a while longer, after the interface is below the effluent port, to begin wasting. The amount of supernatant wasted will be equal to the total volume fed during the sequence. The movement and directional flow of fluid throughout components of the system dunng the feeding and wasting periods is summarized by the arrows in Figure 12.

B. Experimental Protocol

1. Substrate

The organic loading of the feed stream was provided by low heat non fat dry milk (NFDM)¹⁵. The milk was used because it is easily diluted with tap water to produce the desired feed strength, could be stored in bulk for long periods of time, and already contained some essential nutrients. The low heat type milk was used because it contained a higher nitrogen content. The NFDM has a COD value of 1.04

¹⁵Purchased from J M. Swank Company, West LIberty, Iowa 52776.

Figure 12. Schematic diagram of the overall setup for two systems showing the direction of fluid movement

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g COD/g NFDM and other properties similar to those reported by Chiang [2] which are shown in Table 1.

Also added to the feed stream was sodium bicarbonate and a mineral solution. The sodium bicarbonate was added to provide adequate alkalinity levels needed for buffering the system. The amount of sodium bicarbonate added was based on the pH of the system and varied with the HRT and the organic loadmg rate. It was desired to maintain the pH in the 6.8 to 7.2 range.

Trace minerals were added to supplement the milk in order to provide the necessary nutrients required for bacterial growth. The mineral stock solution is shown in Table 2. This recipe is the same to that used in Chiang's research [2], and was shown to be adequate for anaerobic growth associated with the NFDM. The amount of minerals added varied with the organic load and the HRT. Generally, 2 ml of mineral stock solution were added for each g of COD/L/day added, at a HRT of 2.17 days. A change in the amount of mineral solution added was inversely proportional to a change in HRT.

The substrate was prepared in batches of 14 liters. Twelve liters of tap water were added to a 20 liter plastic container. The proper amounts of NFDM, sodium bicarbonate, and minerals were mixed with two liters of water in a household blender¹⁷. These two liters were then added to the 12 liters of tap water, and then the plastic container was agitated to mix the contents. The containers were stored in a

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¹⁷ Hamilton Beach, 14 Speed Blender

Parameter	Values	Units	Reference
COD	1.04	g/g NFDM	This study
BOD ₅	0.49	g/g NFDM	This study
TOC	0.21	g/g NFDM	$[2]$
TKN	5.4	$g/100 g$ NFDM	$[2]$
$T-PO4$	2.2	$g/100 g$ NFDM	$[2]$
Fat	≤ 1.0	$g/100 g$ NFDM	Swiss Valley ¹⁶
Lactose	51.0	$g/100 g$ NFDM	Swiss Valley
Protein	>36.0	$g/100 g$ NFDM	Swiss Valley
Particle Size	98% thru #40 sieve		Swiss Valley
Ash	8.2	$\%$	Swiss Valley
Solub. Index	\leq 1.25	$\%$	Swiss Valley
Std. Plate Bacterial Count	50,000	counts/g NFDM	Swiss Valley
Trace Minerals Fe Ni $\mathbf C$ Mo Zn	4.6 1.0 0.8 3.0 15.0	ppm of NFDM ppm of NFDM ppm of NFDM ppm of NFDM ppm of NFDM	$[2]$ $[2]$ $[2]$ $[2]$ $[2]$

Table 1. Properties of NFDM

¹⁶ Swiss Valley Farms Co., Davenport, Iowa 52808

Chemical Compound	Quantity	Criteria
FeCl ₂ ·4H ₂ O	35.60 g/L	$Fe/NFDM = 200$
ZnCl ₂	2.08 g/L	$Zn/NFDM = 20$
NiCl ₂ ·6H ₂ O	4.05 g/L	$Ni/NFDM = 20$
CoCl ₂ ·6H ₂ O	4.04 g/L	$Co/NFDM = 20$
MnCl ₂ ·4H ₂ O	3.61 g/L	$Mn/NFDM = 20$

Table 2. Recipe for mineral stock solution

refrigerator at about 40 C while the substrate was being pumped out of them.

2. Start up

Once the reactor and components were assembled, the reactor was calibrated in one-half liter increments, by marking on the side of the reactor next to the manometer. The pumps were also calibrated to determine the rough volume of fluid moved per minute at various positions on the pump potentiometer. This calibration is only rough, because as the pump tubing wears the rate of fluid movement changes. Therefore, during operation the volume fed is checked regularly.

The next step of start up was to check for leaks. This was necessary for two reasons, 1) to keep it strictly an anaerobic system, and 2) since the system is under pressure from the level differences in the displacement columns, any leak will decrease the biogas production measurement. In order to check for leaks, the reactor was filled with water to its full position, the gas recycle pump was turned on, and the displacement columns situated as to put a back pressure on the system. In this situation, if the levels in the displacement columns change, there is a leak. The leak was then found by applying a soap solution to joints and connections, while looking for bubbles. Leaks in plexiglas joints were glued and other leaks were fixed with silicone caulking. It was found out early that the less connectors and joints there were, the better, in terms of gas leak prevention.

Once all the leaks were fixed, the reactor was ready to be seeded. The initial seed was obtained from a SMAR which had been previously used in the laboratory for the degradation of NFDM. However, after more problems with leaks and other unforeseen minor incidents an active seed sludge from the anaerobic digesters at the Neveda, Iowa Water Pollution Control Plant (WPCP) was used.

Initial successful operation prompted the set up of a second system. The second reactor was seeded with a digesting sludge from a primary digester at the Ames, Iowa, WPCP. Problems again with the first system resulted in another reseeding. This time the first reactor was also seeded with sludge from the Ames WPCP.

Each time seed was obtained, it was passed through a one mm mesh screen. The seed sludge was then diluted to a solids concentration of about 6,000 mg/L. A peristaltic pump was used to pump the seed into the reactor.

Once a reactor was seeded, the system was not mixed or fed for about six hours to allow facultative organisms to use any available oxygen. Then the reactor was mixed for two or three minutes once every hour to move oxygen out of other areas in the system (tubing, foam separation bottle, etc.). One day after initial seeding, the substrate was fed in low strength batches, of less than 0.5 g/L/day. After gas production was observed on the gas meter, the substrate feed sequence was started at the desired loading and the rest of the operational sequence was implemented.

3. Experimental operation

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a. Phase I The first phase of the research was to determine if an efficient sequential batch operation was possible under anaerobic conditions. This study was accomplished by examining the system at three different HRTs with a nominal loading rate of 1.5 g COD/L/day. The HRTs were 13 days, 2.17 days, and 1 day. The effective volume of the reactors was 13 liters. The contents of the reactors were mixed on a continuous basis from the beginning of the first feeding period of the sequence to the beginning of the settling period. The gas mixing rate used was about one liter/minute, which was enough to gently roll the mixed liquor. The sequence information for each HRT is shown in Table 3.

The temperature of the reactors was maintained at 35° C in a constant temperature room. The mixed liquor concentration of the reactors was allowed to fluctuate as influenced by the COD loading rate and the HRT. The success of this initial study led to the implementation of the remaining research.

b. Phase II In order for the ASBR system to be most beneficial and economical in a real world situation, it was felt that low HRTs would be most applicable. Therefore, in the second phase, three HRTs of 0.54, 1.08, and 2.17 days were examined at loading rates ranging from nominal values 0.5 g COD/L/day to 5.0 g COD/L/day. The non-even HRT length resulted from two factors. One being that the

aFeeding periods lasted for two minutes and were once every other hour with the first period beginning at the beginning of the sequence.

bThe wasting period began 20 minutes prior to the end of the sequence.

cThe settling period began 110 minutes prior to the end of the sequence.

best reactor operation volume was 13 liters, and the other being that the best volume fed and wasted per sequence was 4 liters. As one can tell, using these two numbers one can not come up with a nice even number for a HRT.

The upper loading rates were determined by reactor performance. The maximum load possible, up to failure, was the high load limit for each HRT. The lower loading rates were chosen arbitrarily based on performances in the initial portion of the research. Table 4 shows the loadings and HRTs examined.

The temperature, active volume, and gas mixing rate were the same as in phase 1. The mixed liquor concentration was again allowed to fluctuate. The mixing was continuous for the first three COD loading rates at the 1.08 day HRT. Intermittent mixing was used for the rest of the COD/HRT combinations. The intermittent mixing consisted of 2.5 minutes of mixing every half hour, beginning with the start of the first feeding period and ending 57.5 minutes prior to the end of the sequence.

The rest of the sequence information is shown if Table 5. The time location of feeding periods, which are descnbed in the footnotes of Table 5, provide for 124 minutes of elapsed time from the end of the last feeding period to the beginning of the settling period, for each HRT sequence.

	Loading Rate, g COD/L/Day*						
HRT, days	0.5	1.0	1.5	2.0	3.0	4.0	5.0
0.54	$\mathbf X$	X	X	X	X	NE	
1.08	$\mathbf X$	X	X	X	X	X	NE
2.17	$\bf X$	X	X	X	\mathbf{X}		NE

Table 4. COD loadings and HRTs examined

*A "X" indicates COD data determined at pseudo equilibrium, "NE" indicates data determined before pseudo equilibrium was reached.

		HRT, days	
Sequence Characteristic	0.54	1.08	2.17
Number of sequences per			
day	6	3	1.5
Length of sequence, hrs	$\overline{4}$	8	16
Volume wasted per day, liters	24	12	6
Volume wasted per sequence, liters	4	4	4
Number of feeding periods per sequence	2 ^a	4 ^b	8c
Length of wasting period, minutesd	13	13	13
Length of settling period, minutese	37.5	37.5	37.5

Table 5. Sequence information for phase II

aFeeding periods lasted 11 minutes with the first period beginning at the start of the sequence and the last period beginning 85 minutes after the start of the sequence.

bFeeding periods last 5.5 minutes with the first period beginning at the start of the sequence and the second period beginning 90.5 minutes after the start of the sequence. The third and fourth feeding periods began 4 hours after the first and second periods respectively.

CFeeding periods lasted 12 minutes with the first period beginning at the start of the sequence and the second penod beginning 84 minutes after the start of the sequence. The third, fifth, and seventh feeding periods followed the first period in four hour increments. The remaining periods followed the second period also in four hour increments.

dThe wasting period began 20 minutes prior to the end of the sequence.

eThe settling period began 57.5 minutes prior to the end of the sequence.

c. Other research The final portion of the research looked at the COD reduction and biogas production throughout a sequence. This was accomplished by taking samples throughout the sequence and performing COD analysis on them, and by measuring the biogas production at several periods during the sequence. Also, in relation to this experiment, the relative biogas production throughout a sequence was compared for continuous mixing and intermittent mixing. Table 6 summaries the times throughout the sequence in which tests and measurements were performed.

4. Monitoring analyses

The performance of each system was characterized by several parameters. The minimum frequency for running the total range of parameters was one set of total analyses for each loading at a given HRT. However, some of the tests were run more frequently. The number of repetitions for a given parameter for one data set will be further described below.

a. Chemical oxygen demand The Chemical Oxygen Demand (COD) test was one of the main parameters for determining reactor efficiency. The COD test is a measure of the oxygen equivalent of the organic matter in a sample that is susceptible to oxidation by a strong chemical oxidant. In this test, potassium dichromate is used as the oxidant. The

Table 6. Sequence information for COD reduction and gas production study

Measurement Information, times of measurement

test was used to compare the COD of the feed stream to that of the waste streams, and to monitor the COD value of the mixed liquor in the reactor. The waste stream was analyzed in two different ways. The first, labeled the "effluent stream," consisted of a representative sample taken from the entire wasted batch. The second, labeled the "supernatant stream," consisted of a sample taken halfway down in the settled portion of the supernatant in the reactor. The difference in these two streams being that the effluent stream will contain more solids and theoretically a higher COD value. The reason for this is that a significant amount of solids were trapped in the reactor side ports as the solids settled. When withdrawal of liquid began, solids from the ports flowed out in the liquid stream and were part of the collected effluent. The supernatant sample, on the other hand, was not affected significantly by these trapped solids. Therefore, the effluent stream results correspond to the overall system methane production and COD reduction while the supernatant results are more indicative of a real application that will not have ports where solids can be trapped.

The COD procedure followed was Standard Method 508 B, Oxygen Demand (Chemical), Closed Reflux, Titrimetric Method [37]. The digestion vessel used was the 20 x 150 mm culture tube¹⁸, which required the following quantities:

• Sample	5 ml
• Potassium Dichromate	3 ml
• Sulfuric Acid Reagent	7 ml

¹⁸ Cat. No. 14-9571, Fisher Scientific Company.

Thus, giving a total volume of 15 ml. These volumes allowed for a maximum measuring capacity of 480 mg $O₂/L$. Therefore, some samples required dilution with distilled water to meet this requirement.

The COD was calculated by the following equation [37]:

COD as mg
$$
O_2/L = (A-B) \times M \times 8000 \times DF
$$

ml of sample

where,

The tubes were sealed for digestion with teflon screw caps¹⁹. The tubes were cleaned after each use by scrubbing with soap and water, rinsing six times with tap water and rinsing six times with distilled water, to help prevent organic matter contamination. To prevent leaking, new caps were used for each run.

Generally, for each reactor, four samples were taken for a COD run: 1) feed stream sample, 2) mixed liquor sample, 3) effluent stream sample, and 4) supernatant sample. For each sample a total COD (TCOD) and a soluble (SCOD) were determined. The TCOD was run on the sample as taken, and the SCOD was determined on the filtrate of the

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¹⁹ Cat No. 02-883-8E, Fisher Scientific Company.

sample which passed through a 9 cm GFA glass filter paper²⁰ (a vacuum filter apparatus with buchner funnels was used to aid in filtering the samples). Each total and soluble COD was run in duplicate, thus for one run, on one reactor, 16 COD values were determined.

The method used for determining a data point based on COD, involved running three sets of CODs within a 5-day period, with the reactors at pseudo equilibrium, and averaging the three numbers. The exception to this was at the high loading rates, when the SRT was below the minimum SRT of 10 days. When this happened, only 1 or 2 runs were conducted so that the load could be decreased before system failure occurred. Therefore, these runs were performed at a nonpseudo equilibrium.

b. Solids analysis The solids analysis was the other primary parameter for determming reactor efficiency. Solids analyses were performed at a minimum of once per COD loading/HRT data pomt and was quite often run several times in between. Total and volatile suspended solids were performed according to Standard Methods 209 C and 209 D [37], respectively, except for the following modifications:

209 C

- Filters were not washed prior to weighing. Instead blanks were used to determined mass changes.
- A 10 ml sample size was used throughout the research

²⁰ Cat No. 09-804-90A, Fisher Scientific Company.

- Only one 10 ml washing was conducted.
- Only one cycle of drying, cooling, desiccating, and weighing was utilized.

209 D

• Only one cycle of drying, cooling, desiccating, and weighing was utilized.

The same type of filter paper was used for solids as was used for the COD analysis. Aluminum planchet weighing dishes were used to hold the filter paper. Solids were run on the mixed liquor, the effluent stream, the supernatant, and on blanks. Each sample was run in triplicate with two to four blanks being run. The following equations were used to determine the total and volatile suspended solids (TSS and VSS).

TSS, mg/L =
$$
\frac{(A-B+C) (1000 mg/g) (1000 ml/L)}{\text{sample volume, ml}}
$$

where,

 $A = Weight of filter + planet + dried residue, g,$ $B = Weight of filter + planet, g,$

 $C = Weight loss of blanks, g.$

VSS, mg/L =
$$
\frac{(A-D+C-E) (1000 mg/g) (1000 ml/L)}{\text{sample volume, ml}}
$$

where,

 $A = Wt$. of filter + planchet + residue before ignition, g, $C = Wt$. loss of blanks before ignition, g,

 $D = Wt$. of filter + planchet + residue after ignition, g, $E = Wt$. loss of blanks after ignition, g.

c. Gas analysis The biogas produced by the system was normally analyzed twice a week, but sometimes only once a week. The analysis was performed using a Gas Chromatograph (GC). The GC column used for the analysis detected relative proportions of N_2 , CH₄, and $CO₂$. Calibration of the GC system was accomplished using a custom-made gas standard²¹ which contained 5% N_2 , 70% CH₄, and 25% $CO₂$. The specifications of the GC are shown in Table 7.

The sample for analysis was removed from the previously described gas sampler by inserting a 1 ml syringe22 fitted with a side port needle23 into the gas sampler septum and drawing out the biogas. The first two samples removed were discarded and the third sample was used for the analysis. A 0.9 ml sized sample was then injected into the GC column as soon as possible after it was taken from the sampler. Each run was done in duplicate.

The gas analysis was essential for determining methane production of the reactors and was also useful for determining if any air was entering the system. Evidence of air would be shown by a higher than normal percentage of nitrogen gas.

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²¹ Union Carbide Industrial Gases, Inc., Specialty Gas, East Chicago, Indiana.

²² Hamilton Gastight #1001TLL Syringe, Hamilton Co, Reno, Nv.

²³ Alltech, Side Bore, 22 Gauge Needle, Alltech Associates Inc, Deerfield, Ill

Item	Specification
Gas Chromatograph	Hewlett Packard 5730A
Column	6 ft x 0.125 in I. D., stainless steel
Packing	Porapak Q, 80/100 mesh size
Temperature	Ambient
Carrier gas	Helium
Flowrate	30 ml/min
Detector	Thermal conductivity
Temperature	200° C
Injector block temperature	100° C
Sample size	0.9 ml
Data Station	Maxima data station

Table 7. GC operating parameters for biogas analysis

d. pH The pH of the reactors was determined several times a week on the average, using a Altex pH meter²⁴. The meter was calibrated periodically using buffers of pH 7.00 and 10.00 and calibrated daily using a single buffer solution of pH 7.00.

Samples, approximately SO ml, were taken during the wasting periods of the reactors. The pH was determined immediately after sampling to prevent minimal error due to $CO₂$ release. The pH probe was washed with distilled water and stored in a buffer solution of pH 4.00.

As mentioned before, the pH determination aided in figuring the amount of sodium bicarbonate added. The pH is also helpful in quickly identifying a "sick" reactor. A drop in pH usually means something has inhibited the methane forming bacteria, thus causing an increase in volatile acids and a decrease in pH.

e. Volatile acids The total volatile aCIds were determmed a minimum of one time per COD loadmg/HRT data point. The procedure used was that of Standard Method *S04* B [37], with the followmg exceptions:

- A recovery factor of 0.7 was assumed as suggested in a previous edition of the Standard Method 504 B [36].
- The heating apparatus used, only permitted a distillation rate of approximately *2.S* ml/min.

²⁴ Altex pH Meter, Model 4500, Digital.
The titration to pH 8.3 was performed with the aid of the Altex pH Meter as opposed to using an indicator.

A sample size of about 150 ml was obtained from the effluent stream as the reactors were wasting. Generally, the samples were free of solids enough that centrifuging was not necessary. The sample preparation and subsequent distillation was started as soon as possible after the sample was taken.

The following equation was used to calculate the total volatile acids [37]:

Volatile acids as acetic acid, mg/L = ml NaOH x N x 60,000 ml of sample x f

where,

 $N =$ normality of NaOH,

 $f =$ recovery factor (assumed to be 0.7).

f. Alkalinity The total alkalinity was also determined once per COD loading/HRT data point. The procedure used was that of Standard Method 403 [37]. The titration on the 25 ml sample was completed to an endpoint of 4.3. The sample was taken from the effluent streams of the reactors and the alkalinity test was then run immediately.

The following equation was used to determine the total alkalinity of the sample [37]:

Total Alkalinity, mg $CaCO₃/L = A \times N \times 50,000$ ml sample

where,

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 $A = ml$ standard acid used,

 $N =$ normality of standard acid.

A summary of the monitoring analyses parameters is shown in Table 8.

5. System monitoring and maintenance

a. Monitoring The monitoring and daily system requirements involved several steps which are summarized in Table 9. For the 0.54 and 1.08 day HRTs, the feed was made up every day. The feed for the 2.17 day HRT was made up every other day. Also the gas reading, feed pump calibration, and reactor minimum level check, were conducted every other day at the 2.17 day HRT.

The barometric pressure, room temperature and gas reading were used to calculate the gas volume produced at standard temperature and pressure (STP). The pressure and temperature over a previous one day period were determined by averaging the current day's pressure and temperature with the previous day's pressure and temperature. This step is shown in the following equation used to determine the gas production at STP:

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Parameter	Method	Frequency	Repetitions
Chemical Oxygen Demand	Standard Method 508 B [37]	Once per data Three point (min.)	
Total Suspended Solids	Standard Method 209 C [37]	Once per data point (min.)	Two
Volatile Suspended Solids	Standard Method 209 D [37]	Once per data point (min.)	Two
Biogas Analysis	GC	Bi-weekly	Two
pH	pH meter	Regularly	One
Total Alkalinity	Standard Method 403 [37]	Once per data point (min.)	One
Volatile Acids	Standard Method 504 B [37]	Once per data point (min.)	One

Table 8. Summary of monitoring analyses parameters

Table 9. Monitoring and daily system requirements

- 1. Measure volume of remaining substrate after the last feeding period.
- 2. Adjust pump potentiometer according to volume of remaining substrate.
- 3. If excess feed is remaining, feed to correct volume.
- 4. Clean out containers used to hold feed.
- 5. Record barometer reading and check temperature of the hot room.
- 6. Calibrate pH meter if pH is to run.
- 7. Record gas meter readings after the wasting period has begun.
- 8. Determine pH of samples.
- 9. Mix up new batch of feed, adjust sodium bicarbonate if necessary, based on the pH.
- 10. Turn the effluent pumps on/off to achieve the proper minimum level inside the reactor.
- 11. Adjust the wasting period time on the timer accordingly, based on the results of number 10.

$$
G_{STP} = \frac{(P1 + P2)}{2P_S} \times \frac{T_S}{273.16 + (T_1 + T_2)/2} \times (V_2 - V_1)
$$

where,

$$
G_{\text{STP}} = \text{Gas production at standard pressure and}\n \text{temperature},
$$

 P_1 = Barometric pressure of previous day, mm of Hg,

$$
P_2
$$
 = Barometric pressure of current day, mm of Hg,

$$
P_S
$$
 = Standard pressure = 760 mm of Hg,

$$
T_S
$$
 = Standard temperature = 273.16 °C,

$$
T_1
$$
 = Temperature of previous day, ^oC,

$$
T_2
$$
 = Temperature of current day, ^oC,

$$
V_1
$$
 = Gas meter reading of previous day, liters,

$$
V_2
$$
 = Gas meter reading of current day, liters.

b. Maintenance The maintenance of the system consisted mainly of changing pump tubing and cleaning feed and effluent lines. The schedule followed for the maintenance procedure is shown in Table 10. A1so included in the general maintenance concept was the cleamng and picking up of the hot room and work area.

Item	Frequency
1. Change feed pump tubing	Two weeks
Change gas recycle pump tubing 2.	Two weeks
Change effluent pump tubing 3.	Two months
Change cooling pump tubing 4.	Two months
5. Clean out feed lines	Two weeks
6. Clean out effluent lines	Two months

Table 10. Maintenance schedule

V. RESULTS AND DISCUSSION

A. System Design

The general reactor and system design were unchanged throughout the research and proved to be quite proficient. However, the initial operation necessitated some minor changes to improve the capabilities of the system compared to that described in the Experimental Set-up section.

One of the changes involved the addition of the foam separation bottle. The combination of the milk and sodium bicarbonate in the feed produced foam when mixed in the reactor (the foam was also produced when the feed was mixed in the feed containers). The produced foam would then recirculate with the biogas through the diffuser and, if there were solids in the foam, these solids would plug the inside of the diffuser. The foaming problem also seemed to depend on reactor maturity or performance. The foaming decreased as the reactor contents aged during Phase I (reactor performance was also increasing at the time) to the point of very little foaming during most of the Phase II research.

Another system alteration involved the enlargement of the displacement columns. The size of the first columns allowed only for the displacement of three liters of reactor volume at a time. The new columns were designed large enough to allow for displacement of the total volume of the reactor. The larger columns thus allowed for greater flexibility in terms of sequence operation.

Other minor changes included:

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- The addition of the manometer on the side of the reactor.
- The raising of the gas recycle pump to allow for consolidated liquid in the tubing to flow back into the reactor.
- The relocation of the feed pumps so that the substrate could be pushed through the cold water jacket instead of pulled. This reduced foam and bubbles in the feed which allowed for more accuracy in the volume of feed pumped from day to day.
- The reinforcing of the side ports with the 2.54 cm x 2.54 cm x 0.64 cm (1 in x 1 in x 0.25 in) plexiglas plates. Experience showed that the ports standing alone would break off quite easily.

B. Initial Operation

The period after seeding involved the weeding out of the poorer settling suspended solids as well as the dissolved solids. A solids/supernatant interface would form after the first day, but the supernatant was quite dark due to high amounts of dissolved solids. Depending on the HRT, this dark supernatant would usually clear up in about one to two weeks after seeding. The early loss of solids suggests that it is better to seed at a solids concentration higher than what is desired for the beginning operation of the reactor.

The previously described procedure for depleting the initial oxygen concentration after seeding seemed to be adequate. There were no signs of low gas production or drop in pH which might indicate inhibition due to oxygen presence.

c. Phase I

Two reactors were used throughout Phase I and Phase II of the research. The reactor used to determine the individual data points and the dates the data were obtained and given in the Appendix, Section A. The complete set of data throughout the entire research is shown in the remaining sections in the Appendix. The results presented for Phase I and Phase II can thus be derived from the information in Section A of the Appendix and the data in the other Appendix sections.

The analyses parameters, for determining the results of Phase I as well as Phase II, were performed once a system reached pseudo steady-state. Pseudo steady-state is assumed to be reached when the gas production is constant. This point was generally reached within three HRTs after constant feeding was established.

The loadings and results of Phase I are shown in Table 11. The COD reductions, both soluble and total, were almost all in the 90% range with the exception of the 1.0 day HRT value at 86.7%. The suspended solids values were actually somewhat high compared with some of the values obtained at similar loads in Phase II of the research. The reason for this may have been that all the poorer settling solids had been flushed from the system by the start of Phase II.

	HRT, days		
Parameter	1.0	2.17	13
Actual Organic Loading ^a TCOD, g/L/day SCOD, g/L/day	1.454 1.292	1.291 1.154	1.343 1.290
Suspended Solids Loading TSS, mg/L VSS, mg/L	4407 3815	4442 3777	6589 4326
F/M _b	0.33	0.29	0.20
Effluent COD Total, g/L/day Soluble, $g/L/day$	0.194 0.053	0.111 0.037	0.099 0.048
% COD Reductions Total, % Soluble, %	86.7 95.9	91.4 96.8	92.6 96.2
Effluent Suspended Solids TSS, mg/L VSS, mg/L	282 195	203 125	848 677
Sludge Age	16	47	101
pH	6.84	6.87	7.15
Alkalinity, mg/L as $CaCO3$	1136	2209	4200
Volatile Acids, mg/L Acetic	31	22	80
Gas Production (STP), L	5.6	5.4	6.0
% Methane	74.6	71.6	62.9

Table 11. Phase I loadings and results

aNominal COD loading for all HRT's is 1.5 g TCOD/L/day.

 bF/M = Food to microorganism ratio or energy level and is equivalent to the TCOD loading, g/L/day divided by the VSS, g/L.

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The overall Phase I results showed that the ASBR definitely had the potential to be an efficient process and warranted further research.

D. Phase II

The matrix of COD loading rates and HRTs provided ample combinations for showing the results of Phase II. The three main types of results shown will be COD removal rates, COD removal efficiency, and suspended solids concentrations in the waste stream.

The total and soluble COD removal rates at the various loadings for each HRT are shown in Figures 13, 14, and 15, the data of which are shown in Tables 12, 13, and 14. (Note: As mentioned in Section IV.AA.a., the effluent represents the entire wasted sample including suspended solids trapped in the side ports, while the supernatant is a sample from the settled portion of the reactor.) The plots show that the removal rate is directly proportional to the COD loading except for the highest loading on each of Figures 14 and 15. These points represent the failure of the reactors due to high solids and thus high total COD in the effluent. The soluble removal is still proportional at these loadings indicating that the system is not over saturated in terms of removing COD, but is most likely producing too much gas for good solids settling.

The same COD removal rate data from Tables 12, 13, and 14 can can also be plotted against I/HRT (I/HRT is a common representation for plotting HRT related data for each nominal loading rate). These

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Total COD Loading, g/L/day

Figure 13. COD removal rate at various loading rates, for the 0.54 day HRT

Total COD Loading, g/L/day

Figure 14. COD removal rate at various loading rates, for the 1.08 day HRT

Total COD Loading, g/L/day

Figure 15. COD removal rate at various loading rates, for the 2.17 day HRT

		COD removal rate
Total COD Load g/L/day	Soluble Effluent g/L/day	Total Effluent g/L/day
0.47	0.34	0.35
1.03	0.86	0.69
1.49	1.26	1.20
1.84	1.65	1.43
3.01	2.51	2.05
3.82	3.40	2.72

Table 12. COD removal rate at various loading rates for the 0.54 day HRT

	COD removal rate		
Total COD Load g/L/day	Soluble Effluent g/L/day	Total Effluent g/L/day	
0.46	0.42	0.40	
0.90	0.84	0.81	
1.31	1.23	1.20	
1.72	1.65	1.58	
2.68	2.55	2.55	
3.72	3.55	3.49	
4.68	4.19	3.63	

Tablé 14. COD removal rate at various loading rates for the 2.17 day HRT

plots are shown in Figures 16 and 17. Figure 16 shows the soluble COD removal rate to be independent of the HRTs represented. This is also true for total COD removal at the lower loading rates, shown in Figure 17, but not true at the higher loading rates. The removal rates generally decrease with shorter HRTs at the higher loading rates. This indicates that as the system is pushed, in terms of lower HRTs and higher loading rates, the total COD removal rates tend to decline.

The COD removal efficiency compares the influent COD with the effluent and supernatant COD and is determined in terms of both soluble and total removal. Figures 18, 19, and 20 show the removal efficiencies at various loadings for each HRT. The data for these graphs are shown in Tables 15, 16, and 17 (the tabular data can help distinguish between the soluble supernatant and effluent curves which in some cases are almost on top of each other). The soluble curves tend to increase at first and then level off. At the lower loading rates the soluble loading was increasing but the SCOD of the waste stream was remaining constant, thus the increase in efficiency. Then the SCOD of the waste stream began to increase slowly, but not fast enough to drop the removal efficiencies. The total removal efficiency curves also tended to increase to a point but then begin to fall off. The reason for this can be explained by examining the waste stream solids concentration shown in Figures 21, 22, and 23 (data for which are given in Tables 18, 19, and 20). In each figure there is a loading rate where the curves begin to rise at a faster rate. The loading rate corresponds to the fall of the total COD removal efficiencies curve.

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Figure 16. Soluble COD removal rates at the three HRTs, for all the nominal loading rates

Figure 17. Total COD removal rates at the three HRTs, for all the nominal loading rates

Figure 18. COD removal efficiency at various loading rates, for the 0.54 day HRT

Total COD Loading, g/L/day

Figure 19. COD removal efficiency at various loading rates, for the 1.08 day HRT

Total COD Loading, g/L/day

Figure 20. COD removal efficiency at various loading rates, for the 2.17 day HRT

	COD removal efficiency			
Total COD Load g/L/day	Soluble Supernatant %	Soluble Effluent %	Total Supernatant %	Total Effluent %
0.47	87.7	86.7	77.0	73.8
1.03	92.3	92.3	81.8	71.6
1.49	94.5	94.6	81.3	80.4
1.84	96.4	96.0	83.3	78.0
3.01	95.2	94.6	73.8	68.2
3.82	95.0	94.7	75.0	71.2

Table 15. COD removal efficiency at various loading rates for the 0.54 day HRT

Table 16. COD removal efficiency at various loading rates for the 1.08 day HRT

	COD removal efficiency			
Total COD Load g/L/day	Soluble Supernatant %	Soluble Effluent %	Total Supernatant %	Total Effluent %
0.46	97.3	97.0	90.6	88.6
0.90	98.4	98.3	92.8	89.7
1.31	98.8	98.7	93.0	91.0
1.72	98.6	98.6	95.2	91.9
2.68	98.9	98.9	95.9	95.2
3.72	98.6	98.6	94.8	93.7
4.68	98.2	97.8	86.9	77.5

Table 17. COD removal efficiency at various loading rates for the 2.17 day HRT

1/HRT, 1/days

Figure 21. Total suspended solids concentration of the waste stream at various loadings rates, for the 0.54 day HRT

Total COD Loading, g/L/day

Total suspended solids concentration of the waste stream Figure 22. at various loadings rates, for the 1.08 day HRT

Total COD Loading, g/L/day

Figure 23. Total suspended solids concentration of the waste stream at various loadings rates, for the 2.17 day HRT

Total COD Load g/L/day	Total Supernatant Waste Stream mg/l	Suspended Solids Concentration Effluent Waste Stream mg/l
0.47	35	48
1.03	66	85
1.49	102	125
1.84	142	188
3.01	406	458
3.82	455	527

Table 18. Total suspended solids concentration in the waste streams at various loading rates for the 0.54 day HRT

Total COD Load g/L/day	Total Supernatant Waste Stream mg/l	Suspended Solids Concentration Effluent Waste Stream mg/l
0.46	47	56
0.90	144	193
1.31	168	224
1.72	158	198
2.68	212	225
3.72	350	410
4.68	1064	2225

Table 20. Total suspended solids concentration in the waste streams at various loading rates for the 2.17 day HRT

Thus, the solids increase causes the total COD of the waste stream to increase at a faster rate than the COD loading rate and therefore, the decrease in efficiency. As mentioned before, the effluent and supernatant stream solids most likely increase due to the increase gas production and the general pushing of the system.

Figures 24 through 30 again show the COD removal efficiency, this time at the three HRTs, with each figure representing a different loading rate (plotting data are shown in Tables 15, 16, and 17). In every plot except for one, the COD removal efficiency decreases as the HRT decreases, indicating that as the system is pushed, in terms of HRT, the COD removal efficiency tends to decrease.

Figures 31 through 37 represent sohds concentrations of the waste stream at all three HRTs (data for the plots contained in Tables 18, 19, and 20). Figures 38 and 39 summarize the curves of Figures 31 through 37. When these figures are compared with the previously mentioned graphs of FIgures 21, 22, and 23, there is evidence that suspended solids is related more to loading rate than HRT. Figures 21, 22, and 23 show suspended solids increasing with load while Figures 38 and 39 show no outright relationship with HRT. This helps support the theory that the increase gas production from the higher loads decreases the settleability of solids. The settling efficiency decrease could also be related to the higher energy level from increased feed strength causing poorer flocculation and coagulation, as predicted by McKinney [27]. Figure 40, representing effluent suspended solids concentration compared to F/M (food to microorganism ratio, with the

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COD removal efficiency at the three HRTs, for the nominal Figure 24. loading rate of 0.5 g TCOD/L/day

Total COD Loading, g/L/day

COD removal efficiency at the three HRTs, for the nominal Figure 25. loading rate of 1.0 g TCOD/L/day

COD removal efficiency at the three HRTs, for the nominal Figure 26. loading rate of 1.5 g TCOD/L/day

Figure 27. COD removal efficiency at the three HRTs, for the nominal loading rate of 2.0 g TCOD/L/day

COD removal efficiency at the three HRTs, for the nominal Figure 28. loading rate of 3.0 g TCOD/L/day

Figure 29. COD removal efficiency at the three HRTs, for the nominal loading rate of 4.0 g TCOD/L/day

Figure 30. COD removal efficiency at two HRTs, for the nominal loading rate of 5.0 g TCOD/L/day

1/HRT, 1/days

Figure 31. Total suspended solids concentration of the effluent and supernatant at all three HRTs, for the nominal loading rate of 0.5 g TCOD/L/day

1/HRT, 1/days

Figure 32. Total suspended solids concentration of effluent and supernatant at all three HRTs, for the nominal loading rate of 1.0 g TCOD/L/day

1/HRT, 1/days

Total suspended solids concentration of effluent and Figure 33. supernatant at all three HRTs, for the nominal loading rate of 1.5 g TCOD/L/day

1/HRT, 1/days

Figure 34. Total suspended solids concentration of the effluent and supernatant at all three HRTs, for the nominal loading rate of 2.0 g TCOD/L/day

1/HRT, 1/days

Figure 35. Total suspended solids concentration of the effluent and supernatant at all three HRTs, for the nominal loading rate of 3.0 g TCOD/L/day

1/HRT, 1/days

Figure 36. Total suspended solids concentration of the effluent and supernatant at all three HRTs, for the nominal loading rate of 4.0 g TCOD/L/day

1/HRT, 1/days

Figure 37. Total suspended solids concentration of the effluent and supernatant at two HRTs, for the nominal loading rate of 5.0 g TCOD/L/day

Figure 38. Total suspended solids concentration of the supernatant at all three HRTs, for all the nominal loading rates

Figure 39. Total suspended solids concentration of the effluent at all three HRTs, for all the nominal loading rates

Figure 40. Total suspended solids concentration of the effluent at various food to microorganism ratios, for all three HRTs

microorganisms being represented by volatile solids, so that F/M is equivalent to COD loading rate divided by volatile suspended solids of the mixed liquor) or energy level, shows a general trend of solids increasing with increasing F/M. However, as the graph shows, an increase in F/M does not automatically indicate an increase in suspended solids concentration.

As with effluent suspended solids, COD removal efficiency also can not be directly related to F/M , as shown in Figure 41. There is a general trend of decreased efficiency with higher F/M, but no direct relationship is apparent.

As stated in the Experimental Protocol section, the mixed liquor and thus, the solids retention time were allowed to fluctuate as influenced by the HRT and COD loading rate. The total and volatile suspended solids concentrations at the various loading rates for each HRT are given in Tables 21, 22, and 23 and shown in Figures 42, 43, and 44. The mixed liquor tended to increase as the loading rate increased, but dropped off as the solids concentration in the effluent increased. The SRT values given in Table 24 and shown in Figure 45 also indicate a drop off at higher loading rates to the point of failure (SRT less than 10 days) at the 0.54 and 1.08 day HRT.

The data for pH, total alkalinity, and total volatile acids at various loading rates are shown in Figures 46, 47, and 48, respectively. An attempt was made to maintain pH 7.0 by changing the amount of sodium bicarbonate added. This, in turn, affected the total alkalinity of

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Figure 41. Total COD removal efficiency of the effluent at various food to microorganism ratios, for all three HRTs

	Suspended		Solids Concentration	
Total COD Load g/L/day	Volatile mg/1		Total mg/l	
0.47	2834		3440	
1.03	3705		4428	
1.49	6528		7616	
1.84	7880		9102	
3.01	8999		10368	
3.82	6081	6951		

Table 21. Mixed liquor total and volatile suspended solids concentration at various loading rates for the 0.54 day HRT

	Suspended	Solids Concentration		
Total COD Load g/L/day	Volatile mg/l	Total mg/l		
0.46	5424	6502		
0.90	6194	7296		
1.31	6475	7594		
1.72	8226	9614		
2.68	10255	11927		
3.72	10847	12527		
4.68	11198	13192		

Table 23. Mixed liquor total and volatile suspended solids concentration at various loading rates for the 2.17 day HRT

Total COD Loading, g/L/day

Figure 42. Mixed liquor suspended solids concentration at various loading rates, for the 0.54 day HRT

Total COD Loading, g/L/day

Figure 43. Mixed liquor suspended solids concentration at various loading rates, for the 1.08 day HRT

Total COD Loading, g/L/day

Figure 44. Mixed liquor suspended solids concentration at various loading rates, for the 2.17 day HRT

	Solids retention time, days				
Nominal COD Load g/L/day	HRT, days 0.54	HRT, days 1.08	HRT, days 2.17		
0.5	39	62	252		
1.0	28	16	82		
1.5	33	12	73		
2.0	26	46	105		
3.0	12	58	115		
4.0	7	16	66		
5.0		5	13		

Table 24. Solids retention times at various loading rates for HRTs of 0.54, 1.08, and 2.17 days

Figure 45. Solids retention time at various loading rates, for all three HRTs

Total COD Loading, g/L/day

Figure 46, pH of the effluent at various loading rates, for all three **HRTs**

Total alkalinity of the effluent at various loading rates, for all three HRTs Figure 47.

Total COD Loading, g/L/day

Total volatile acids of the effluent at various loading rates, Figure 48. for all three HRTs

the system. The total volatile acids were quite low throughout the research, except at some of the higher loading rates.

The percentage methane in the biogas produced at the various loading rates for each HRT are shown in Figure 49. For each HRT the methane percentage generally decreases as the loading rate and stress on the methane formers increases.

Figure 50 shows the methane gas production at various loading rates for the 2.17 day HRT. The theoretical values are calculated from the total COD removal rates and the relationship that 0.35 L of methane are produced for each gram of total COD reduced. As can be seen, the theoretical values are increasingly further apart from the measured values as more biogas is produced. This is due to the nature of the displacement columns. Up to one liter of biogas can be released to the gas meter in a matter of seconds. This results in inaccurate measurements in the total amount of produced biogas.

E. Other Results

The results of the experiment described in Section IV.B.3.C. (which involved COD and biogas production measurement throughout a sequence) are shown in Figures 51 and 52. The soluble curve shown in Figure 51 has the general appearance of a decay curve, flattening out near the end of the cycle. This indicates that the cycle length provided enough time in terms of COD removal but may still have been too short

Figure 49. The percentage methane of the produced biogas at various loading rates, for all three HRTs

Total COD Loading, g/L/day

Figure 50. Methane production at various loading rates, for the 2.17 day HRT

Figure 51. Supernatant COD values as measured throughout a sequence

Sequence time, minutes

Figure 52. Biogas production as measured throughout a sequence

in terms of releasing sufficient amounts of attached biogas for efficient solids settling.

The biogas production curves in Figure 52 are of the same general shape but the continuous mixing curve is significantly higher. This probably means one of two things. Either the continuous mixing was able to release some unreleased biogas from the intermittent mixing or the continuous mixing is a more efficient method of mixing.

Intermittent mixing was first looked into after the continuous gas mixing continued to cause problems by wearing out pump tubing in a matter one or two days. Biogas production was compared over several days, and showed no significant difference between the two types of mixing. Therefore, the switch was made to intermittent mixing. The question presented now concerns the comparison of continuous and intermittent mixing. Hopefully this will be answered in current and future research.

Other fundamental observations and discussion of the research include:

• Although identifying specific microorganisms in the mixed liquor was not a part of the research, Figures 53 and 54 show typical microorganisms and floc matrices present in the mixed liquor.

Figure 55 shows a sample of mixed liquor immediately after removal from a reactor. On the right is a sample showing how clear the effluent is at low solids concentrations. Figure 56 shows the developed solids/supernatant interface of the same mixed liquor sample 15

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Figure 53. Picture of microorganisms and floc matrix in the mixed liquor

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Figure 54. Picture of microorganisms and floc matrix in the mixed liquor

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Figure 55. Picture of mixed liquor (on the left) immediately after removal from a reactor, and a sample of effluent (on the right)

Figure 56. Picture of mixed liquor (on the left) 15 minutes after removal from the reactor, and a sample of effluent (on the right)

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minutes after removal from the reactor. The solids/supernatant interface as it appears in the reactor is shown in Figure 57.

As the mixed liquor developed the settled volume decreased for the same suspended solids concentration. This would indicate that a denser floc might be developing as the reactor contents age. In the same volume needed to settle solids of the 4000 mg/L range, near the beginning of the research, 12,000 mg/L solids were settling at the end of the research.

There was a limit for this reactor configuration on the amount of suspended solids that would settle. At 15,000 mg/L and a TCOD loading rate of 4.0 $g/L/day$, the mixed liquor would break into two sections during the settling period. One section of solids would float to the top while the other section would settle to the bottom, leaving a clear space in between. Upon decreasing the solids concentration while retaining the same load, the mixed liquor immediately began to settle again in one section.

Large changes in feed concentration can cause settling problems when the mixed liquor concentration is relatively low. At one point the loading rate was doubled from 1.5 to 3.0 g COD/L/day at a suspended solids concentration of around 4000 mg/L. The system began loosing solids rapidly and could not recover, leading to failure. However, slower increases at higher suspended solids concentration resulted in little increase in waste stream solids.

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Figure 57. Picture of the solids/supernatant interface during settling in the reactor

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VI. ENGINEERING APPLICATION

A. Design Example

An ASBR example design will be performed on a wastewater from FDL Foods in Dubuque, Iowa. This design will be compared to the actual design for the wastewater which consisted of a covered anaerobic lagoon [12]. The design will be conducted in "English units" for easier comparison to the actual design, but "SI" units will also be shown for the major design parameters.

The design values for the wastewater are as follows:

Flow = 2.832 mgd $(10,715 \text{ m}^3/\text{day})$ $BOD_5 = 68,275$ lb/day (30,969 kg/day)

The operating volume of the reactor will be based on the design loading rate of 0.10 lb BOD_5 /ft³/day (1.60 g $BOD_5/L/day$) which is conservatively chosen based on the ASBR research.

> $V = (68,275 \text{ lb/day})/(0.10 \text{ lb } BOD_5/\text{ft}^3/\text{day})$ $V = 682,750$ ft³ (19,322 m³)

In order to help compare respective areas of the two systems, a design depth of 28 feet will be selected (the FDL Foods anaerobic lagoon also has a depth of 28 feet).

$$
A = (682,750 \text{ ft}^3)/(28 \text{ ft})
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$$
A = 24,384 \text{ ft}^2 \qquad (2265 \text{ m}^2)
$$

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$$
A = 0.56 \text{ acres}
$$

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The hydraulic retention time of the ASBR equals:

HRT = $(682,760 \text{ ft}^3)(7.48 \text{ gal/ft}^3)/(2,832,000 \text{ gal/day})$ $HRT = 1.8$ days

A design assumption of three sequence periods per day gives the volume of wastewater processed per sequence to be 944,000 gals (3572 m^3) . The wastewater will be fed continuously for the first three hours of the sequence. Therefore, assuming a continuous flow rate, a holding basin would be required to hold five hours of wastewater flow, until the next feeding period of the next sequence.

The minimum design volume of this holding basin would be:

 $V = (2,832,000 \text{ gals/day})(5 \text{ hrs/day})/(7.48 \text{ gals/ft}^3)$ $V = 78,877 \text{ ft}^3$ (2232 m³)

The actual basin size would need to be designed based on the peak wastewater flow of the plant for any given consecutive 5 hours.

The mixing of the system would consist of a five minute mixing period every 30 minutes beginning with the start of the sequence and ending after the 10th mixing period (6 hours and 45 minutes into the sequence). After this last mixing period the settling period would begin and last for 40 minutes, The remaining 35 minutes of the 8 hour sequence would involve decanting the supernatant.

The minimum depth of the supernatant/solids interface is based on the reactor area and the amount of wastewater processed per sequence. This depth is calculated as follows:

D =
$$
(944,000 \text{ gals})/(7.48 \text{ gals/ft}^3)/(24,384 \text{ ft}^2)
$$

D = 5.2 ft (1.6 m)

Actually, the interface should be somewhat lower than 5.2 ft before wasting to prevent settled solids from being drawn out of the system with the decanted supernatant. But, even if the interface needed to be 7 ft below the surface, the solids still would only need to settle to 75% of the total depth.

The methane production and heating value can be determined based on an estimated value for BOD reduction and the relationship that 5.61 ft³ of methane is produce per lb of BOD ultimate (BOD_L) destroyed (@ STP).

> Assume a value of 1.5 for the ratio of BOD_L to BOD_S Assume a conservative value of 85% BOD_L reduction

 BOD_L lbs reduced per day:

 $=$ (1.5 BOD_I/BOD5)(68,275 lb BOD₅/day)(0.85) $= 87,050$ lbs BOD_L/day (39,485 kg BODL/day)

Standard volume of methane produced per day:

- $=$ (87,050 lbs BOD_L/day)(5.61 ft³/lb BOD_L)
- $= 488,000 \text{ ft}^3/\text{day}$ @ STP (13,820 m³/day)

Heating value of gas (assuming methane has a heating value of 1000 BTU per ft^3 :

=
$$
(488,000 \text{ ft}^3 \text{ CH}_4/\text{day})
$$

= $\frac{488 \text{ million BTU/day}}{488 \text{ million BTU/day}}$ (143,000 kWh/day)

The simplest and likely the most inexpensive design would involve a covered lagoon type system for the reactor. This would take advantage of the extra insulation provided by the ground and the flexibility in the cover would allow for the necessary volume displacement during the feeding and wasting periods. Mixing would be accomplished by pulling the gas off the top of the reactor and pumping it though a header system that feeds several diffusers located in the bottom of the lagoon. The excess gas would be collected and used for fuel in the plant or for heating the contents of the lagoon.

The holding basin could also be of the covered lagoon nature, the contents of which would be continuously gas mixed. The mixing would benefit stabilization and also provide for uniform feed concentration to the main reactor.

B. Design Comparison/Summary

Table 25 summarizes the ASBR design and also shows the comparison to the actual FDL design. The big value that jumps out of the comparison is the significant difference in the total lagoon volume. This much difference in volume would provide significant cost savings

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Table 25. Comparison of design parameters for FDL foods wastewater

which would more than offset the extra costs needed for mixing and effluent pumping equipment.

The FDL ASBR design might be compared to the research conducted to try and estimate the systems efficiency. If the 1.5 value assumption of the BOD_L to BOD_S ratio is applied to the BOD_S loading rate, it gives a BOD_L loading of 2.40 g/L/day. One could then approximate the COD loading to be about 2.40 $g/L/day$. This would fall well within the range for efficient COD removals shown by this research. As long as this ratio does not run much higher than about 2.0 the research shows the system should perform in the 90% COD (BOD) removal range.

The sequence parameters for the ASBR design were chosen without knowledge of the FDL wastewater peak flow periods or daily plant operation duration. In an actual ASBR application the feeding period should be adjusted to occur simultaneously with the peak flow. An example of this would be a situation where a peak flow would occur 6 times a day for one hour. In this situation, 4 sequences might be used with the feeding period lasting at least the duration of the peak flow. This type of strategy would significantly reduce the volume required for the holding lagoon. If the plant only operates 8 or 16 hours a day, then one sequence per day would be used. The feeding period would then run the duration of the work day and the rest of the day (sequence) would then be used for mixing, reacting, and wasting. This would completely eliminate the holding basin.

VII. CONCLUSIONS/SUMMARY

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The ASBR system design and successful introductory operation provided the means to further investigate the ASBR. The Phase I results showed that high COD removal efficiencies were possible and that an anaerobic system could accomplish internal settling.

Phase II involved loading the new process to the point of failure at three HRTs: 0.54, 1.08, and 2.17 days. The shortest HRT was able to effectively remove COD up to a 3.0 g TCOD/L/day loading rate before dropping below the minimum 10 day SRT at the 4.0 $g/L/day$ loading. The other two HRTs were able to efficiently reach a 4.0 $g/L/day$ loading rate. At the 5.0 g/L/day loading, the 1.08 day HRT failed due to a SRT below minimum, the 2.17 day HRT did not actually fail at this loading rate due to SRT, but when the data were collected the system had not reached pseudo steady-state. Failure was inevitable, though, because the effluent solids concentration was about 2250 mg/L .

One of the other important aspects learned about the ASBR operation concerned the maximum obtainable mixed liquor concentration. For this reactor configuration the mixed liquor would not settle properly at concentrations around 15,000 mg/L. The highest effective mixed liquor concentration was in the 12,000 to 13,000 mg/L.

Another important operational concept involved load mcreases. Unless the system is at high mixed liquor concentrations, the loading should rate should be increased slowly, in increments of about 0.2 to 0.4 g/L/day.

The bottom line of the research is that in its simplest mode of operation, the ASBR was able to effectively perform internal settling while removing low to medium strength organic loads at short HRTs.

Thus, an anaerobic system can be successfully operated without using external settling and degasification or requiring media to maintain significant SRTs.

VIII. RECOMMENDATIONS FOR FURTHER RESEARCH

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The initial studies of the ASBR have been conducted at low to medium levels of organic loads $(0.5 \text{ to } 5.0 \text{ g/L/day})$ at low HRTs $(0.54,$ 1.08, and 2.17 days), with 1.5 to 6 sequences per day. These parameters provide the means to prove that the ASBR is a viable process for industrial wastewater treatment. However, more work needs to be done to further examine the fundamentals of the ASBR operation. Currently, other research on the ASBR is being conducted at Iowa State University in the areas of reactor configuration and thermophilic temperatures. Research at ISU has also been conducted on swine wastes using the ASBR at temperatures of 250 C and 350 C [9] and on a high strength (60 g COD/L), high solids biotechnology waste. Some of the other areas that need further research include:

1) *Effects of the timing of the feeding perIOd dUring the sequence.* The time during the sequence in which feeding occurs could be very significant to the efficiency of the system. The key is to have the most degradation possible prior to the settling period. Does this happen if all the feed for the sequence is fed quickly at the beginning of the period, slowly throughout the period, or in two or more batches, as was the case in this research?

2) *Comparison of intermittent mixing with continuous mixing.* Theoretically there seems to be benefits to both. Intermittent mixing

utilizes less energy but may not allow enough microorganism contact. However, does continuous mixing provide too much shearing force in terms of removing food attached to the biomass? Also, how does the mixing scheme affect the attachment of gas bubbles to the biomass? These are some of the questions that need to be answered in terms of the best type or combination of mixing.

3) Settling times of the sludge/supernatant interface. The mixed liquor content, organic loading rate, and possibly the reactor configuration have important roles in determining the settling rate of the interface. The question is, "Is there a relationship between these parameters and are there other parameters that can be used to predict the settling time of the interface?"

4) *Effect of granulation*. How difficult is it to develop and/or maintain a granulated sludge and how beneficial is this type of mixed liquor in terms of reactor efficiency?

5) Overall stability of the system. How well does the system handle shock loads or varying organic loads and HRTs, and what can be done from a design or operational standpoint to minimize the effects of load variations.

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APPENDIX

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A. Information for COD Loading/HRT Data Points

			Period Data Obtained		
Nominal COD Loading Rate g/L/Day	HRT days	Reactor Used R1, R2	Beginning Date	Ending Date	
1.5	1.00	R ₂	07/19/90	07/25/90	
1.5	2.17	R ₂	07/09/90	07/16/90	
1.5	13.00	R1	07/09/90	07/19/90	

Phase I Information for COD Loading/HRT Data Points

Nominal			Period Data Obtained		
COD Loading		Reactor	Beginning	Ending	
Rate	HRT	Used	Date	Date	
g/L/Day	days	R1, R2			
0.5	0.54	R ₂	11/19/91	11/21/91	
0.5	1.08	R ₂	10/22/91	10/24/91	
0.5	2.17	R1	12/17/91	12/19/91	
1.0	0.54	R ₂	12/10/91	12/13/91	
1.0	1.08	R ₂	10/02/91	10/05/91	
1.0	2.17	R ₁	01/07/91	01/11/91	
1.5	0.54	R ₂	01/07/91	01/11/91	
1.5	1.08	R ₂	09/21/91	09/25/91	
1.5	2.17	R ₁	01/21/91	01/25/91	
2.0	0.54	R ₂	01/21/91	01/25/91	
2.0	1.08	R ₂	02/04/91	02/06/91	
2.0	2.17	R1	02/04/91	02/06/91	
3.0	0.54	R ₂	02/11/91	02/13/91	
3.0	1.08	R1	02/11/91	02/13/91	
3.0	2.17	R1	02/18/91	02/21/91	
4.0	0.54	R ₂	03/11/91	03/13/91	
4.0	1.08	R ₂	02/18/91	02/20/91	
4.0	2.17	R1	02/27/91	02/27/91	
5.0	1.08	R ₂	02/27/91	02/27/91	
5.0	2.17	R ₁	03/07/91	03/09/91	

Phase II Information for COD Loading/HRT Data Points

 \hat{A}

B. COD Data

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Phase I COD Data (feed stream)

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 $\hat{\mathcal{A}}$

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Phase I COD Data (mixed liquor and waste streams)

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Phase I COD Data (mixed liquor and waste streams)

Phase II COD Data (feed stream)

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Phase II COD Data (feed stream)

Phase II COD Data (mixed liquor and waste streams)

Phase II COD Data (mixed liquor and waste streams)

Phase II COD Data (mixed liquor and waste streams)

C. Solids Data

			Reactor 1							
			Effluent Mixed Liquor				Supernatant			
Days	Date	TSS	VSS	TSS	VSS	TSS	VSS			
		mg/L	mg/L	mg/L	mg/L	mg/L	mg/L			
$\mathbf{1}$	06/20/90			16360	9530					
$\overline{\mathbf{3}}$	06/22/90	14455	8277	12918	7803					
6	06/25/90	12918	7302	1335	822					
8	06/27/90	10575	6188	1388	868					
10	06/29/90	9440	5768	870	578					
13	07/02/90	8080	4977	507	360					
20	07/09/90	6973	4507	850	650					
23	07/12/90	6300	4183	810	633					
24	07/13/90	6493	4287	803	633					
30	07/19/90	5590	3863	923	793					
35 ₂	07/24/90	6230	4363	1283	1110					
36	07/25/90			1110	960					

Phase I Solids Data

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Phase II Solids Data

		Reactor ₂						
		Mixed	Liquor		Effluent		Supernatant	
Days	Date	TSS	VSS	TSS	VSS	TSS	VSS	
		mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	
295	02/06/91	10910	9343	197	137	200	140	
300	02/11/91	10227	8863	503	377	453	330	
	302 02/13/91	10510	9135	413	295	360	235	
307	02/18/91	9747	8447	680	527	633	523	
309	02/20/91	9145	7960	607	425	567	428	
	314 02/25/91	9625				960		
315	02/26/91	10595				2370		
316	02/27/91	9225	7980	2150	1817	1287	1007	
328	03/11/91	6992	6120	592	472	612	432	
329	03/12/91	6750	5875	493	340	377	233	
330	03/13/91	7112	6248	495	375	375	300	

Phase II Solids Data

D. Total Gas Production Data

		Reactor 1		Reactor ₂			
Days	Date	Last Rdg days	Std. Gas Volume liters	Days	Date	Last Rdg days	Std. Gas Volume liters
$\mathbf{1}$	06/20/90	$\mathbf{1}$	2.79	38	05/25/90	$\mathbf{1}$	8.32
$\boldsymbol{2}$	06/21/90	$\mathbf{1}$	6.73	39	05/26/90	$\mathbf{1}$	7.96
3	06/22/90	$\mathbf{1}$	8.06	40	05/27/90	$\mathbf{1}$	8.15
$\boldsymbol{4}$	06/23/90	$\mathbf{1}$	8.94	41	05/28/90	$\mathbf{1}$	8.14
5	06/24/90	$\mathbf{1}$	8.85	42	05/29/90	$\mathbf{1}$	7.97
6	06/25/90	$\mathbf{1}$	9.12	43	05/30/90	$\mathbf{1}$	7.92
$\boldsymbol{7}$	06/26/90	$\mathbf{1}$	9.46	44	05/31/90	$\mathbf{1}$	7.30
8	06/27/90	$\mathbf{1}$	9.88	45	06/01/90	$\mathbf{1}$	8.95
9	06/28/90	$\mathbf{1}$	10.47	46	06/02/90		
10	06/29/90	$\mathbf{1}$	11.49	47	06/03/90	$\overline{2}$	10.82
11	06/30/90	$\mathbf{1}$	9.94	48	06/04/90	$\mathbf{1}$	6.50
12	07/01/90	$\mathbf{1}$	9.72	49	06/05/90	$\mathbf{1}$	7.02
13	07/02/90	$\mathbf{1}$	8.80	50	06/06/90	$\mathbf{1}$	6.05
14	07/03/90			51	06/07/90	$\mathbf{1}$	6.33
15	07/04/90			52	06/08/90	$\mathbf{1}$	6.24
16	07/05/90			53	06/09/90	$\mathbf{1}$	6.25
17	07/06/90			54	06/10/90	$\mathbf{1}$	6.32
18	07/07/90			55	06/11/90	$\mathbf{1}$	6.45
19	07/08/90			56	06/12/90	$\mathbf{1}$	10.14
20	07/09/90	$\mathbf{1}$	7.92	57	06/13/90	$\mathbf{1}$	3.66
21	07/10/90		8.75	58	06/14/90	1	6.68
22	07/11/90	$\mathbf{1}$	9.77	59	06/15/90	$\mathbf{1}$	7.29
23	07/12/90	$\mathbf{1}$	9.19	60	06/16/90	$\mathbf{1}$	6.93
24	07/13/90	$\mathbf{1}$	9.55	61	06/17/90		
25	07/14/90			62	06/18/90	$\overline{2}$	14.48
26	07/15/90	$\boldsymbol{2}$	19.81	63	06/19/90	$\mathbf{1}$	6.85
27	07/16/90	$\mathbf{1}$	9.69	64	06/20/90	$\mathbf{1}$	5.13

Phase I Gas Production Data

		Reactor ₁		Reactor ₂				
Days	Date	Last Rdg days	Std. Gas Volume liters	Days	Date	Last Rdg days	Std. Gas Volume liters	
				92	07/18/90	$\mathbf{1}$	5.67	
				93	07/19/90	$\mathbf{1}$	5.42	
				94	07/20/90	$\mathbf{1}$	6.01	
				9 ₅	07/21/90	$\mathbf{1}$	5.82	
				96	07/22/90			
				97	07/23/90	$\overline{2}$	10.67	
				98	07/24/90	$\mathbf{1}$	5.91	
				99	07/25/90	$\mathbf{1}$	5.57	
				100	07/26/90	$\mathbf{1}$	5.53	
				101	07/27/90	$\mathbf{1}$	14.18	
				102	07/28/90	$\mathbf{1}$	12.49	
				103	07/29/90	$\mathbf{1}$	12.43	
				104	07/30/90	$\mathbf{1}$	12.09	
				105	07/31/90	$\mathbf{1}$	11.88	

Phase I Gas Production Data

		Reactor ₁		Reactor ₂			
Days	Date	Last Rdg days	Std. Gas Volume liters	Days	Date	Last Rdg days	Std. Gas Volume liters
43	08/01/90	$\mathbf{1}$	19.69	106	08/01/90	$\mathbf{1}$	6.20
44	08/02/90	$\mathbf{1}$	19.08	107	08/02/90	$\mathbf{1}$	4.52
45	08/03/90	$\mathbf{1}$	19.51	108	08/03/90	$\mathbf{1}$	5.87
46	08/04/90	$\mathbf{1}$	19.50	109	08/04/90	$\mathbf{1}$	5.65
47	08/05/90	$\mathbf{1}$	16.85	110	08/05/90	$\mathbf{1}$	5.44
48	08/06/90	$\mathbf{1}$	16.97	111	08/06/90	$\mathbf{1}$	5.19
49	08/07/90	$\mathbf{1}$	18.09	112	08/07/90	$\mathbf{1}$	5.13
50	08/08/90	$\mathbf{1}$	18.97	113	08/08/90	$\mathbf{1}$	5.87
51	08/09/90	$\mathbf{1}$	18.75	114	08/09/90	$\mathbf{1}$	5.36
52	08/10/90	$\mathbf{1}$	19.08	115	08/10/90	$\mathbf{1}$	5.36
53	08/11/90	$\mathbf{1}$	19.78	116	08/11/90	$\mathbf{1}$	4.91
54	08/12/90	$\mathbf{1}$	20.05	117	08/12/90	$\mathbf{1}$	6.29
55	08/13/90	$\mathbf{1}$	19.39	118	08/13/90	$\mathbf{1}$	5.81
56	08/14/90	$\mathbf{1}$	20.08	119	08/14/90	$\mathbf{1}$	5.43
57	08/15/90	$\mathbf{1}$	19.05	120	08/15/90	$\mathbf{1}$	5.61
58	08/16/90	$\mathbf{1}$	19.32	121	08/16/90		
59	08/17/90	$\mathbf{1}$	18.75	122	08/17/90	$\overline{2}$	7.96
60	08/18/90	$\mathbf{1}$	18.79	123	08/18/90	$\mathbf{1}$	0.36
61	08/19/90	$\mathbf{1}$	18.28	124	08/19/90	$\mathbf{1}$	5.65
62	08/20/90	$\mathbf{1}$	17.73	125	08/20/90	$\mathbf{1}$	3.69
63	08/21/90	$\mathbf{1}$	16.82	126	08/21/90	1	3.25
64	08/22/90	$\mathbf{1}$	17.38	127	08/22/90	$\mathbf{1}$	3.45
65	08/23/90	$\mathbf{1}$	19.13	128	08/23/90	$\mathbf{1}$	2.66
66	08/24/90	$\mathbf{1}$	12.46	129	08/24/90	$\mathbf{1}$	2.63
67	08/25/90	$\mathbf{1}$	13.14	130	08/25/90	$\mathbf{1}$	2.59
68	08/26/90	$\mathbf{1}$	14.40	131	08/26/90	$\mathbf{1}$	2.87
69	08/27/90	$\mathbf{1}$	14.20	132	08/27/90	$\mathbf{1}$	3.34

Phase II Gas Production Data

Phase II Gas Production Data

		Reactor ₁		Reactor ₂			
Days	Date	Last Rdg days	Std. Gas Volume liters	Days	Date	Last Rdg days	Std. Gas Volume liters
97	09/24/90	$\mathbf{1}$	20.67	160	09/24/90		
98	09/25/90	$\mathbf{1}$	19.21	161	09/25/90	$\overline{2}$	2.98
99	09/26/90	$\mathbf{1}$	19.27	162	09/26/90	$\mathbf{1}$	1.71
100	09/27/90	$\mathbf{1}$	19.46	163	09/27/90	$\mathbf{1}$	0.22
101	09/28/90	$\mathbf{1}$	18.99	164	09/28/90	$\mathbf{1}$	0.09
102	09/29/90	$\mathbf{1}$	18.38	165	09/29/90		
103	09/30/90	$\mathbf{1}$	18.50	166	09/30/90	$\overline{2}$	1.07
104	10/01/90	$\mathbf{1}$	17.63	167	10/01/90		
105	10/02/90	$\mathbf{1}$	18.65	168	10/02/90	$\overline{2}$	1.00
106	10/03/90	$\mathbf{1}$	18.96	169	10/03/90	$\mathbf{1}$	0.16
107	10/04/90	$\mathbf{1}$	17.50	170	10/04/90	$\mathbf{1}$	0.54
108	10/05/90	$\mathbf{1}$	18.55	171	10/05/90	$\mathbf{1}$	0.36
109	10/06/90	$\mathbf{1}$	17.43	172	10/06/90	$\mathbf{1}$	0.37
110	10/07/90	$\mathbf{1}$	18.00	173	10/07/90	$\mathbf{1}$	0.18
111	10/08/90	1	18.31	174	10/08/90	$\mathbf{1}$	0.26
112	10/09/90	\mathbf{I}	18.36	175	10/09/90	$\mathbf{1}$	0.23
113	10/10/90	$\mathbf{1}$	18.15	176	10/10/90	$\mathbf{1}$	0.36
114	10/11/90	$\mathbf{1}$	18.48	177	10/11/90	$\mathbf{1}$	0.39
115	10/12/90	$\mathbf{1}$	18.03	178	10/12/90	$\mathbf{1}$	0.57
116	10/13/90	$\mathbf{1}$	18.07	179	10/13/90	$\mathbf{1}$	0.75
117	10/14/90	$\mathbf{1}$	16.77	180	10/14/90	$\mathbf{1}$	0.78
118	10/15/90	$\mathbf{1}$	16.64	181	10/15/90	$\mathbf 1$	0.77
119	10/16/90	$\mathbf{1}$	18.04	182	10/16/90	$\mathbf{1}$	0.41
120	10/17/90	$\mathbf{1}$	18.26	183	10/17/90	$\mathbf{1}$	0.00
121	10/18/90	$\mathbf{1}$	9.02	184	10/18/90	$\mathbf{1}$	1.00
122	10/19/90			185	10/19/90		
123	10/20/90			186	10/20/90	$\overline{2}$	2.11

Phase II Gas Production Data

Phase II Gas Production Data

Phase II Gas Production Data

		Reactor 1		Reactor ₂				
Days	Date	Last Rdg days	Std. Gas Volume liters	Days	Date	Last Rdg days	Std. Gas Volume liters	
178	12/14/90	$\overline{2}$	3.34	241	12/14/90	$\mathbf{1}$	5.86	
179	12/15/90			242	12/15/90	$\mathbf{1}$	5.91	
180	12/16/90	$\mathbf{2}$	2.51	243	12/16/90	$\mathbf{1}$	6.28	
181	12/17/90			244	12/17/90	$\mathbf{1}$	6.28	
182	12/18/90	$\overline{2}$	4.30	245	12/18/90	$\mathbf{1}$	5.32	
183	12/19/90			246	12/19/90	$\mathbf{1}$	5.83	
184	12/20/90	$\overline{2}$	3.48	247	12/20/90	$\mathbf{1}$	8.72	
185	12/21/90			248	12/21/90	$\mathbf{1}$	9.45	
186	12/22/90			249	12/22/90	$\mathbf{1}$	8.83	
187	12/23/90	3	11.26	250	12/23/90	$\mathbf{1}$	8.79	
188	12/24/90			251	12/24/90	$\mathbf{1}$	8.62	
189	12/25/90	$\overline{2}$	8.18	252	12/25/90	$\mathbf{1}$	7.88	
190	12/26/90			253	12/26/90	$\mathbf{1}$	9.46	
191	12/27/90	$\boldsymbol{2}$	6.63	254	12/27/90	$\mathbf{1}$	8.85	
192	12/28/90			255	12/28/90	$\mathbf{1}$	9.18	
193	12/29/90	$\overline{2}$	10.24	256	12/29/90	$\mathbf{1}$	8.58	
194	12/30/90			257	12/30/90	$\mathbf{1}$	9.06	
195	12/31/90	$\overline{2}$	8.92	258	12/31/90	$\mathbf{1}$	8.38	
196	01/01/91			259	01/01/91			
197	01/02/91	$\overline{2}$	8.58	260	01/02/91	$\overline{2}$	16.55	
198	01/03/91	$\mathbf{1}$	2.68	261	01/03/91	$\mathbf{1}$	10.22	
199	01/04/91			262	01/04/91	$\mathbf{1}$	8.60	
200	01/05/91	$\boldsymbol{2}$	8.93	263	01/05/91			
201	01/06/91			264	01/06/91	$\mathbf{2}$	21.92	
202	01/07/91	$\boldsymbol{2}$	3.44	265	01/07/91	$\mathbf{1}$	10.91	
203	01/08/91			266	01/08/91	$\mathbf{1}$	17.01	
204	01/09/91	$\boldsymbol{2}$	8.09	267	01/09/91	$\mathbf{1}$	19.53	

Phase II Gas Production Data

		Reactor ₁		Reactor ₂			
Days	Date	Last Rdg days	Std. Gas Volume liters	Days	Date	Last Rdg days	Std. Gas Volume liters
205	01/10/91			268	01/10/91	$\mathbf{1}$	16.99
206	01/11/91	$\overline{2}$	7.72	269	01/11/91	$\mathbf{1}$	20.19
207	01/12/91			270	01/12/91	$\mathbf{1}$	21.07
208	01/13/91	$\overline{2}$	10.28	271	01/13/91	$\mathbf{1}$	19.34
209	01/14/91			272	01/14/91	$\mathbf{1}$	20.32
210	01/15/91	$\overline{2}$	12.01	273	01/15/91	$\mathbf{1}$	21.23
211	01/16/91			274	01/16/91	$\mathbf{1}$	22.93
212	01/17/91	$\overline{2}$	12.49	275	01/17/91	$\mathbf{1}$	7.85
213	01/18/91			276	01/18/91	$\mathbf{1}$	25.51
214	01/19/91	$\overline{2}$	10.41	277	01/19/91	$\mathbf{1}$	25.70
215	01/20/91			278	01/20/91	$\mathbf{1}$	27.15
216	01/21/91	$\overline{2}$	13.52	279	01/21/91	$\mathbf{1}$	25.70
217	01/22/91			280	01/22/91	$\mathbf{1}$	17.03
218	01/23/91	$\overline{2}$	10.70	281	01/23/91	$\mathbf{1}$	15.54
219	01/24/91			282	01/24/91	$\mathbf{1}$	14.10
220	01/25/91	$\overline{2}$	9.66	283	01/25/91	$\mathbf{1}$	14.33
221	01/26/91			284	01/26/91	$\mathbf{1}$	15.17
222	01/27/91	$\overline{2}$	12.23	285	01/27/91	$\mathbf{1}$	15.09
223	01/28/91			286	01/28/91	$\mathbf{1}$	9.10
224	01/29/91	$\boldsymbol{2}$	11.46	287	01/29/91	$\mathbf{1}$	10.51
225	01/30/91			288	01/30/91	1	13.17
226	01/31/91	$\overline{2}$	15 44		289 01/31/91	$\mathbf{1}$	11.92
227	02/01/91				290 02/01/91	$\mathbf{1}$	13.34
228	02/02/91	$\boldsymbol{2}$	15.92	291	02/02/91	$\mathbf{1}$	14.57
229	02/03/91				292 02/03/91	$\mathbf{1}$	15.17
230	02/04/91	$\overline{2}$	15.70	293	02/04/91	$\mathbf{1}$	13.90
231	02/05/91			294	02/05/91	$\mathbf{1}$	10.66

Phase II Gas Production Data

		Reactor ₁		Reactor ₂				
Days	Date	Last Rdg days	Std. Gas Volume liters	Days	Date	Last Rdg days	Std. Gas Volume liters	
232	02/06/91	$\overline{2}$	14.45	295	02/06/91	$\mathbf{1}$	8.38	
233	02/07/91			296	02/07/91			
234	02/08/91	$\overline{2}$	17.15	297	02/08/91	$\overline{2}$	16.55	
235	02/09/91	$\mathbf{1}$	9.85	298	02/09/91	$\mathbf{1}$	10.22	
236	02/10/91	$\mathbf{1}$	9.78	299	02/10/91	$\mathbf{1}$	8.60	
237	02/11/91	$\mathbf{1}$	11.10	300	02/11/91			
238	02/12/91	$\mathbf{1}$	11.39	301	02/12/91	$\overline{2}$	21.92	
239	02/13/91	$\mathbf{1}$	11.09	302	02/13/91	$\mathbf{1}$	10.91	
240	02/14/91			303	02/14/91	$\mathbf{1}$	17.01	
241	02/15/91	$\overline{2}$	25.61	304	02/15/91	$\mathbf{1}$	19.53	
242	02/16/91			305	02/16/91	$\mathbf{1}$	16.99	
243	02/17/91	$\overline{2}$	25.66	306	02/17/91	$\mathbf{1}$	20.19	
244	02/18/91			307	02/18/91	$\mathbf{1}$	21.07	
245	02/19/91	$\overline{2}$	26.47	308	02/19/91	$\mathbf{1}$	19.34	
246	02/20/91			309	02/20/91	$\mathbf{1}$	20.32	
247	02/21/91	$\overline{2}$	26.30	310	02/21/91	$\mathbf{1}$	21.23	
248	02/22/91			311	02/22/91	$\mathbf{1}$	22.93	
249	02/23/91	$\overline{2}$	31.36	312	02/23/91	$\mathbf{1}$	7.85	
250	02/24/91			313	02/24/91	$\mathbf{1}$	25.51	
251	02/25/91	$\overline{2}$	35.89	314	02/25/91	$\mathbf{1}$	25.70	
252	02/26/91			315	02/26/91	$\mathbf{1}$	27.15	
	253 02/27/91	$\boldsymbol{2}$	36.50	316	02/27/91	$\mathbf{1}$	25.70	
	254 02/28/91			317	02/28/91	$\mathbf{1}$	17.03	
255	03/01/91	$\overline{2}$	38.63		318 03/01/91	$\mathbf{1}$	15.54	
256	03/02/91			319	03/02/91	$\mathbf{1}$	14.10	
	257 03/03/91	$\overline{2}$	50.64	320	03/03/91	$\mathbf{1}$	14.33	
258	03/04/91			321	03/04/91	$\mathbf{1}$	15.17	

Phase II Gas Production Data

		Reactor ₁		Reactor ₂				
Days	Date	Last Rdg days	Std. Gas Volume liters	Days	Date	Last Rdg days	Std. Gas Volume liters	
259	03/05/91	2	50.29	322	03/05/91	$\mathbf{1}$	15.09	
260	03/06/91			323	03/06/91	$\mathbf{1}$	9.10	
261	03/07/91	$\overline{2}$	46.23	324	03/07/91	$\mathbf{1}$	10.51	
262	03/08/91			325	03/08/91	$\mathbf{1}$	13.17	
263	03/09/91	$\mathbf{2}$	47.05	326	03/09/91	$\mathbf{1}$	11.92	
264	03/10/91			327	03/10/91	$\mathbf{1}$	13.34	
265	03/11/91	$\mathbf{2}$	50.56	328	03/11/91	$\mathbf{1}$	14.57	
266	03/12/91	$\mathbf{1}$	12.20	329	03/12/91	$\mathbf{1}$	15.17	
267	03/13/91	$\mathbf{1}$	2.57	330	03/13/91	$\mathbf{1}$	13.90	
268	03/14/91	1	3.29	331	03/14/91	$\mathbf{1}$	10.66	

Phase II Gas Production Data

E. Gas Analysis Data

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Days	Date	Nitrogen $\%$	Reactor ₁ Carbon Dioxide %	Methane %
7	06/26/90	1.49	34.78	63.73
13	07/02/90	1.53	29.84	68.63
21	07/10/90	3.08	34.29	62.63
24	07/13/90	1.49	35.33	63.18
28	07/17/90	2.10	34.90	62.92
31	07/20/90	1.93	35.56	62.51
35	07/24/90	1.50	36.28	62.22
42	07/31/90	1.04	38.45	60.51

Phase I Gas Analysis

		Reactor ₂			
Days	Date	Nitrogen $\%$	Carbon Dioxide $\%$	Methane $\%$	
$\mathbf{1}$	04/18/90	73.30	10.56	16.15	
9	04/26/90	4.40	37.09	58.51	
16	05/03/90	5.54	31.55	62.90	
31	05/18/90	5.12	36.29	58.59	
50	06/06/90	5.71	28.37	65.92	
55	06/11/90	5.54	26.08	68.38	
63	06/19/90	6.48	28.24	65.29	
70	06/26/90	4.26	27.64	68.10	
76	07/02/90	4.94	20.55	74.51	
84	07/10/90	5.76	25.04	69.20	
87	07/13/90	4.37	22.70	72.93	
91	07/17/90	3.78	23.61	72.61	
94	07/20/90	4.72	20.35	74.93	
98	07/24/90	4.89	18.76	76.35	
105	07/31/90	7.53	27.10	65.37	

Phase I Gas Analysis

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Phase II Gas Analysis

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Phase II Gas Analysis

		Reactor ₂			
Days	Date	Nitrogen %	Carbon Dioxide $\%$	Methane $\%$	
322	03/05/91	1.94	31.10	66.96	
326	03/09/91	3.07	25.03	71.91	
329	03/12/91	3.14	26.58	70.27	
332	03/15/91	4.16	22.53	73.32	

Phase II Gas Analysis

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F. Methane Production Data

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Phase I Methane Production Data

Reactor 1			Reactor ₂		
Days	Date	Methane liters	Days	Date	Methane liters
45	08/03/90	11.92	108	08/03/90	3.95
49	08/07/90	10.92	126	08/21/90	2.26
58	08/16/90	11.59	129	08/24/90	1.92
63	08/21/90	9.95	132	08/27/90	2.50
66	08/24/90	7.11	150	09/14/90	0.41
69	08/27/90	8.12	154	09/18/90	0.17
84	09/11/90	1.69	161	09/25/90	2.23
87	09/14/90	10.72	203	11/06/90	2.43
91	09/18/90	11.61	206	11/09/90	3.03
98	09/25/90	12.58	210	11/13/90	4.13
140	11/06/90	2.07	237	12/10/90	3.47
143	11/09/90	2.87	245	12/19/90	4.32
147	11/13/90	4.16	260	01/02/91	6.37
174	12/10/90	1.73	274	01/16/91	17.44
183	12/19/90	1.33	280	01/22/91	13.03
197	01/02/91	3.02	285	01/27/91	10.54
211	01/16/91	4.17	290	02/01/91	9.46
217	01/22/91	3.51	294	02/05/91	7.61
222	01/27/91	4.11	297	02/08/91	12.29
227	02/01/91	5.17	300	02/11/91	8.19
231	02/05/91	4.75	304	02/15/91	12.90
234	02/08/91	5.81	308	02/19/91	12.57
237	02/11/91	7.45	311	02/22/91	14.96
241	02/15/91	8.09	314	02/25/91	16.12
245	02/19/91	8.27	318	03/01/91	10.21
248	02/22/91	9.57	322	03/05/91	10.10
251	02/25/91	10.89	326	03/09/91	8.57
255	03/01/91	11.67	329	03/12/91	10.66

Phase II Methane Production Data

Reactor 1			Reactor ₂		
Days	Date	Methane liters	Days	Date	Methane liters
259	03/05/91	15.04			
263	03/09/91	14.10			
266	03/12/91	3.90			

Phase II Methane Production Data

G. pH Data

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Phase I pH Data

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Phase I pH Data

Phase II pH Data

Phase II pH Data

Reactor 1			Reactor ₂		
Day	Date	pH	Day	Date	pH
			322 323 325 327 329	03/05/91 03/06/91 03/08/91 03/10/91 03/12/91	7.06 6.86 6.84 6.97 7.00

Phase II pH Data

H. Volatile Acids Data

199

Reactor 1			Reactor ₂		
Days	Date	Volatile Acids mg/L	Days	Date	Volatile Acids mg/L
23	07/12/90	79	86	07/12/90	22
30	07/19/90	81	93	07/19/90	31
36	07/25/90	92	99	07/25/90	43

Phase I Volatile Acid Data

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	Reactor ₁			Reactor ₂	
Days	Date	Volatile Acids mg/L	Days	Date	Volatile Acids mg/L
44	08/02/90	255	107	08/02/90	221
51	08/09/90	184	114	08/09/90	186
56	08/14/90	163	119	08/14/90	99
97	09/24/90	133	150	09/14/90	40
105	10/02/90	129	160	09/24/90	47
182	12/18/90	43	168	10/02/90	47
203	01/08/91	17	189	10/23/90	10
218	01/23/91	60	217	11/20/90	46
231	02/05/91	27	238	12/11/90	43
238	02/12/91	47	266	01/08/91	21
246	02/20/91	27	281	01/23/91	64
253	02/27/91	171	294	02/05/91	29
263	03/09/91	99	301	02/12/91	58
			307	02/18/91	172
			329	03/12/91	51

Phase II Volatile Acid Data

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I. Alkalinity Data

	Reactor 1			Reactor ₂	
Days	Date	Alkalinity mg/L	Days	Date	Alkalinity mg/L
23	07/12/90	4231	86	07/12/90	2209
30	07/19/90	4168	93	07/19/90	1136
36	07/25/90	4105	99	07/25/90	2376

Phase I Alkalinity Data

	Reactor 1			Reactor ₂	
Days	Date	Alkalinity mg/L	Days	Date	Alkalinity mg/L
45	08/03/90	5569	108	08/03/90	2874
56	08/14/90	6886	119	08/14/90	2615
97	09/24/90	4040	150	09/14/90	2300
105	10/02/90	4460	160	09/24/90	2380
126	10/23/90	3560	168	10/02/90	2380
182	12/18/90	1320	189	10/23/90	2240
203	01/08/91	1670	217	11/20/90	750
218	01/23/91	2420	238	12/11/90	1300
231	02/05/91	3570	266	01/08/91	1370
238	02/12/91	3320	281	01/23/91	1800
246	02/20/91	3800	294	02/05/91	2800
253	02/27/91	4200	301	02/12/91	2100
263	03/09/91	4280	307	02/18/91	3540
			329	03/12/91	2440

Phase II Alkalinity Data

