Anaerobic sequencing batch reactor treatment of municipal landfill leachate at 35°C

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Anaerobic sequencing batch reactor treatment of
municipal landfill leachate at 35° C

by

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I. INTRODUCTION

Sanitary landfills have been the primary dumping ground for municipal solid waste for many years. Even today, when concerns over environmental protection are at the forefront of world politics, recent studies indicate that the majority of municipal solid waste generated in the U.S. is still ultimately disposed of in the sanitary landfill [48]. Landfill disposal of municipal solid wastes has continued to be popular because it is one of the simplest and most economical disposal methods available. The use of the sanitary landfill also minimizes adverse environmental effects and other risks and inconveniences, and allows waste to decompose under controlled conditions until its eventual transformation into relatively inert, stabilized material [28].

A. The Leachate Problem

Although the sanitary landfill is the most popular method of solid waste disposal, there are certain environmental hazards associated with landfilling that must be controlled. One of the major hazards is the possible contamination of neighboring surface and/or groundwaters by migrating leachate. In order to prevent the migration of leachate, leachate collection systems, including impermeable liners and drainage pipes, have been developed by the U. S. Environmental Protection Agency and are implemented in today's landfill designs. However, even though it is possible to prevent leachate migration, it is impossible to prevent leachate production, and all of the leachate that is produced at a landfill site must be treated in a safe and effective manner.

Leachates are liquid wastes produced at all landfill sites as water percolates through the refuse and leaches out an assortment of organic and inorganic constituents [1,25,26,28,59,64]. The characteristics of the leachate produced from a sanitary landfill can
vary widely in quantity and composition from site to site and seasonally at each landfill [17]. The factors controlling the composition of the leachate include the degree of compaction and composition of the solid waste, particle size, the hydrology of the site, the climate, and the age of the landfill [28]. The possibility of substantial concentrations of organic materials and various soluble metals, as well as the high level of volume and strength variability, make the treatment of most leachates much more difficult than the treatment of municipal wastewater.

Treatment technologies for landfill leachate are relatively young. Evaluation of various leachate treatment processes was first conducted by Boyle and Ham [1] in 1974. This research provided a foundation for further development in the area of leachate treatment. Since those initial studies, the methods available for the treatment of leachates have expanded dramatically. Table 1 classifies a variety of alternatives available for the partial or total treatment of landfill leachates [28].

Table 1. Alternatives for the treatment of landfill leachates [28]

<table>
<thead>
<tr>
<th>Leachate Channeling</th>
</tr>
</thead>
<tbody>
<tr>
<td>Combined treatment with domestic wastewaters</td>
</tr>
<tr>
<td>Recycling</td>
</tr>
<tr>
<td>Lagooning with recycling</td>
</tr>
<tr>
<td>Biological Processes</td>
</tr>
<tr>
<td>Aerobic treatment</td>
</tr>
<tr>
<td>Anaerobic treatment</td>
</tr>
<tr>
<td>Chemical/Physical Treatment</td>
</tr>
<tr>
<td>Chemical precipitation</td>
</tr>
<tr>
<td>Chemical oxidation</td>
</tr>
<tr>
<td>Adsorption onto activated carbon</td>
</tr>
<tr>
<td>Reverse osmosis</td>
</tr>
<tr>
<td>Ammonia stripping</td>
</tr>
</tbody>
</table>
The variability of the leachate from site to site can create problems in selecting an adequate treatment method. What may be a successful treatment method at one site may not work well at another location. Extensive testing must be conducted to ensure that the best treatment method is selected for each particular leachate.

**B. Objectives and Scope of Study**

The purpose of this study was to examine the feasibility of leachate treatment using a biological process called the anaerobic sequencing batch reactor (ASBR). The ASBR is a new technology which has been developed by Dague and co-workers at Iowa State University. The ASBR is a patented process (U.S. Patent No. 5,185,079).

Phase I of this study was conducted using two identical anaerobic reactors that were fed municipal landfill leachate from the Iowa City, Iowa landfill. These reactors were operated over a range of organic loading rates (OLRs) and hydraulic retention times (HRTs). While operating at each selected set of parameters, the system stability was monitored by performing various laboratory tests. The contents of the reactors were also tested for build up of inorganic particles.

Phase II used the effluent from the ASBR for treatment through an aerobic polishing unit. The aerobic unit consisted of a continuously fed, intermitantly decanted reactor which was operated at a 24 hour HRT. This phase of the study was done to determine whether or not direct discharge limits could be achieved.

The overall objectives of this research can be summarized as:

1. Determining the feasibility of the ASBR for treatment of municipal landfill leachate
2. Identifying if substantial build up of inorganic precipitates hinder reactor performance
3. Determining if an ASBR system with aerobic polishing can achieve direct discharge limits
II. LITERATURE REVIEW

The utilization of anaerobic digestion for waste stabilization goes back nearly one hundred years. The purpose of this section is not to comprehensively cover all aspects concerning the history of anaerobic digestion. Instead, this review will emphasize selected areas of anaerobic digestion. The main areas of concentration include: microbiology and biochemistry, environmental and operational elements affecting anaerobic digestion, the ASBR process, leachate characteristics, and applications of anaerobic treatment to leachate.

A. Microbiology and Biochemistry

The microbiological environment needed for successful anaerobic treatment is complicated. It utilizes various types of anaerobic bacteria which work together to convert complex organics into biogas, which is a combination of carbon dioxide (CO$_2$) and methane (CH$_4$).

In 1964, McCarty [32] described the conversion of complex organics to biogas as a two-stage process. Stage one was defined as the conversion stage during which "acid formers" were able to hydrolyze and ferment the complex organics such as fats, proteins, and carbohydrates into simple organic materials, usually organic fatty acids. In stage two, the "methane formers," which are a consortia of strictly anaerobic bacteria, stabilize the waste by converting the organic acids into biogas.

In more recent work, McInerney et al [37] reported that the conversion process required to anaerobically digest complex organics is actually a three-step process. These three steps have been identified as:

1. hydrolysis and acidogenesis
2. acetogenesis
3. Methanogenesis

Novaes [42] has drawn a typical schematic of the metabolic pathways required for the complete anaerobic conversion of waste to methane (Figure 1). It should be noted that while it is acceptable to describe the anaerobic digestion process in three separate steps, each step is an integral part of the overall waste stabilization process, and is required to ensure complete waste conversion. The major chemical reactions involved in these three steps are shown in Table 2, using glucose as a sample substrate [20]. Although there are three stages identified, there are five major types of anaerobic bacteria needed for complete anaerobic digestion. Table 3 presents typical population densities for the five most common bacterial groups found in anaerobic sludge digesters [54].

1. Hydrolysis and Acidogenesis

This step in the conversion process utilizes the fermentative bacteria. These bacteria alone are not very efficient at recycling organic carbon. Rather, the major function of the fermentative bacteria is the breakdown of biologically synthesized polymers to monomeric units and the conversion of these units to even simpler compounds [12]. The bacteria are able to do this by producing extracellular enzymes such as protease, cellobiase, and amylase [60]. In turn, these enzymes are able to hydrolyze the complex organic matter into simpler soluble organics. These soluble organics are then able to be utilized by the bacteria. Once the fermentative bacteria are able to use the hydrolyzed organics, they can produce end products such as ethanol, acetate, butyrate, and propionate [10,14,42,44,60]. Although the waste is hydrolyzed and used by the fermentative bacteria, there is essentially no stabilization occurring during this stage. The bacteria simply change the form of the waste products so they are able to be stabilized by the other bacteria [32,44]. In order to ensure an environment conducive to the fermentative bacteria, it is important that the temperature, mixing regime, and microorganism population are all closely monitored [44]. These conditions are extremely
Figure 1. Metabolic pathways of anaerobic digestion
Table 2. Thermodynamics of anaerobic digestion [10]

<table>
<thead>
<tr>
<th>Reaction</th>
<th>$\Delta G^\circ$ (kJ/reaction)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(standard conditions)</td>
</tr>
<tr>
<td><strong>Group 1 organisms</strong>&lt;sup&gt;a&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>glucose $\Rightarrow$ 2 acetate+2HCO$_3$+4H$^+$+4H$_2$</td>
<td>-206.3</td>
</tr>
<tr>
<td>glucose $\Rightarrow$ butyrate+2HCO$_3$+3H$^+$+2H$_2$</td>
<td>-254.8</td>
</tr>
<tr>
<td>1.5 glucose $\Rightarrow$ 2 propionate+acetate+3H$^+$+2H$_2$</td>
<td>-465.0</td>
</tr>
<tr>
<td>glucose $\Rightarrow$ 2 ethanol+2CO$_2$</td>
<td>-235.0</td>
</tr>
<tr>
<td><strong>Group 2 organisms</strong></td>
<td></td>
</tr>
<tr>
<td>butyrate $\Rightarrow$ 2 acetate+H$^+$+2H$_2$</td>
<td>+48.1</td>
</tr>
<tr>
<td>propionate $\Rightarrow$ acetate+HCO$_3$+H$^+$+3H$_2$</td>
<td>+76.1</td>
</tr>
<tr>
<td>ethanol $\Rightarrow$ acetate +H$^+$+2H$_2$</td>
<td>+9.6</td>
</tr>
<tr>
<td><strong>Group 3 organisms</strong></td>
<td></td>
</tr>
<tr>
<td>4H$_2$+CO$_2$ $\Rightarrow$ CH$_4$</td>
<td>-135.6</td>
</tr>
<tr>
<td>acetate $\Rightarrow$ CH$_4$+CO$_2$</td>
<td>-31.0</td>
</tr>
<tr>
<td><strong>Overall process</strong></td>
<td></td>
</tr>
<tr>
<td>glucose $\Rightarrow$ 3CH$_4$+3CO$_2$</td>
<td>-393.1</td>
</tr>
</tbody>
</table>

<sup>a</sup>Water left out for brevity
Table 3. Typical population densities for the five groups of bacteria most commonly found in anaerobic sludge digesters [54]

<table>
<thead>
<tr>
<th>Group</th>
<th>Numbers (per ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrolytic bacteria</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>$10^8 - 10^9$</td>
</tr>
<tr>
<td>Proteolytic</td>
<td>$10^7$</td>
</tr>
<tr>
<td>Cellulolytic</td>
<td>$10^5$</td>
</tr>
<tr>
<td>Hydrogen producing</td>
<td></td>
</tr>
<tr>
<td>acetogenic bacteria</td>
<td>$10^6$</td>
</tr>
<tr>
<td>Homoacetogenic bacteria</td>
<td>$10^5 - 10^6$</td>
</tr>
<tr>
<td>Methanogenic bacteria</td>
<td>$10^6 - 10^8$</td>
</tr>
<tr>
<td>Sulfate reducing bacteria</td>
<td>$10^4$</td>
</tr>
</tbody>
</table>

critical if a waste is very complex and difficult to hydrolyze, because poor reaction kinetics can cause the hydrolytic fermentation step to be rate limiting [52]. Even if all of these conditions are optimized, there can still be a portion of the waste which is non-hydrolyzable. This portion of the waste is residual and is carried throughout the treatment process.

2. Acetogenesis

The acetogenic step utilizes two types of bacteria. They are the hydrogen producing acetogenic bacteria and the homoacetogenic bacteria.

a) Hydrogen producing acetogenic bacteria

This group of bacteria are known as obligate proton reducers since their major role is to oxidize fatty acids or alcohols with the reduction of protons to molecular hydrogen [46,54]. The consortia of bacteria included in this group are able to oxidize alcohols such as ethanol to
acetate and H$_2$. They also cause β-oxidation of fatty acids of even numbered carbons to acetate and of fatty acids of odd numbered carbons to acetate, propionate, and H$_2$. The hydrogen producing acetogenic bacteria are also able to perform the decarboxylation of propionate to acetate, CO$_2$, and H$_2$ [46].

b) Homoacetogenic bacteria

Although little is known about the actual role of the homoacetogenic bacteria, they have been classified as chemolithotrophic, H$_2$ and CO$_2$ users [42]. These bacteria have been found to have high thermodynamic efficiencies during their metabolism as a result of no formation of H$_2$ and CO$_2$ during growth on multi-carbon compounds [42]. Whatever the role of homoacetogens, the net result of their metabolism in the anaerobic digestion process is the maintenance of low H$_2$ partial pressures [54].

3. Methanogenesis

There are presently 30 different species within the consortia of methanogenic bacteria which are able to form methane as their metabolic end point [52]. The methanogenic bacteria are the most important within the overall stabilization process because they are the only anaerobic organisms effective at: (1) using electrons in the form of H$_2$ and (2) breaking down acetate anaerobically without exogenous electron acceptors (e.g., nitrate or sulfate) [54]. The characteristics of some commonly studied methanogenic bacteria are shown in Table 4.

While there are many species of methanogenic bacteria, there are very few sources of energy available to them. It is presently believed that only formic acid, acetic acid, methanol, hydrogen, carbon dioxide, and methylamines can be used for both carbon and energy sources by the various species of methanogenic bacteria [54]. The most common energy sources for the methanogenic bacteria are acetate, H$_2$, and CO$_2$. These substrates are utilized by the aceticlastic bacteria and carbon dioxide reducing methanogens, respectively.
<table>
<thead>
<tr>
<th>Organism</th>
<th>Growth substrates</th>
<th>Relevant properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methanobacterium thermoaotrophicum (DH and Marburg strains)</td>
<td>H₂+CO₂</td>
<td>thermophile; rapid growth; high yields</td>
</tr>
<tr>
<td>Methanosarcina barkeri (Schnellen strain)</td>
<td>H₂+CO₂; CH₃OH; acetate; methylamine</td>
<td>mesophile; slow growth; high yields</td>
</tr>
<tr>
<td>Methanococcus thermolithotrophicus</td>
<td>H₂+CO₂; formate</td>
<td>thermophile halophile; rapid growth; very little cell wall; fragile</td>
</tr>
<tr>
<td>Methanobrevibacterium ruminantium (strain M-1)</td>
<td>H₂+CO₂; formate</td>
<td>mesophile; requires exogenous coenzyme M</td>
</tr>
<tr>
<td>Methanococcus vannielii</td>
<td>H₂+CO₂; formate</td>
<td>mesophile; very little cell wall; fragile</td>
</tr>
<tr>
<td>Methanospirillum hungatei</td>
<td>H₂+CO₂; formate</td>
<td>mesophile; sheath largely protein; inner cell wall, probably protein rich polymer; can spheroplast</td>
</tr>
<tr>
<td>Methanobacterium bryantii</td>
<td>H₂+CO₂</td>
<td>mesophile; possible Ni²⁺ bonding in cell wall; can form protoplasts</td>
</tr>
</tbody>
</table>
The acetic acid cleavage reaction utilizes the following equation [32].

\[ \text{C}^*\text{H}_3\text{COOH} \rightarrow \text{C}^*\text{H}_4 + \text{CO}_2 \]

In this reaction, the methyl carbon of acetic acid, marked with an asterisk above, together with its three hydrogen atoms, are converted intact into methane gas [32]. The remaining carbon is converted into carbon dioxide. This reaction accounts for approximately 72% of the methane produced during anaerobic digestion [10,32,44,52].

The carbon dioxide reducing methanogens account for the remaining 28% of the methane production, and follow the reaction below [10,32,44,52].

\[ \text{CO}_2 + 8\text{H} \rightarrow \text{CH}_4 + 2\text{H}_2\text{O} \]

The hydrogen in this reaction is removed from the organic compounds by the extracellular enzymes. The hydrogen is then able to reduce the carbon dioxide to methane gas [32]. This occurs because the carbon dioxide molecules act as hydrogen acceptors.

4. Sulfate Reducing Bacteria

The sulfate reducing bacteria also play an important role in the waste stabilization process, although it is uncertain exactly what that role entails. At typical digester concentrations, the H₂S itself is not toxic to the methane producing bacteria [43]. However, these bacteria are capable of affecting the growth of the methanogens in other ways. There is evidence to support three general relations between the sulfate reducing and methane producing bacteria: (1) competition between the two groups for limited electron donors; (2) coexistence through the use of separate resources; or (3) a synergism in which members of one of the two groups supply an electron donor needed by the other [43].

5. Conclusion

All of the bacteria involved in the anaerobic digestion process are important. However, it is the methanogenic bacteria that are the most critical to the stabilization process. These bacteria are slow growing, and are very susceptible to changes within the digester
environment. It is extremely important to maintain optimum conditions within the digestion system to ensure methane formation. The methanogenic bacteria are the only bacteria capable of effectively degrading and stabilizing organic matter within the anaerobic digester [54]. If the methanogens fail, intermediates such as propionate and butyrate build up in the digester causing the pH to drop and the system to fail [46].

B. Elements Affecting Anaerobic Digestion

There are many parameters involved in the process of anaerobic digestion that must be maintained at optimum conditions to ensure successful treatment. These elements can be separated into two categories: environmental and operational.

1. Environmental Elements
   a) Temperature

   The temperature at which an anaerobic treatment system operates is very important to the overall treatment effectiveness. In a system operating under optimum environmental conditions the most important factor affecting the rate of microbial growth is temperature [9]. The time required for sludge stabilization is directly dependent on the temperature at which the digestion occurs [7]. It has been shown that the rate of biochemical reactions increases with increased heat energy as measured by temperature, as long as the components themselves are unchanged by the heat energy [8]. The temperature has also been noted to affect the rate of synthesis, regeneration, and endogenous respiration [45].

   There have been two optimum temperature ranges identified for anaerobic treatment. They are 30° C to 38° C for the mesophilic bacteria, and 50° C to 60° C for the thermophilic bacteria [44]. Generally speaking, higher operating temperatures result in faster stabilization rates. However, the additional heat requirements often offset any advantage gained due to the
increased cost. Because of this, most biological systems are designed for operation within the mesophilic range, usually at 35°C [8].

There has been extensive testing done on anaerobic systems operating at lower temperatures [11,21,44,45,51]. In a 1927 publication, Rudolphs [51] demonstrated that anaerobic digestion was successful at a temperature of 10°C. As the temperature is lowered, the rates of the biological reactions decrease [8]. This causes the time required for adequate waste stabilization to increase dramatically. It is possible to operate anaerobic systems at lower temperatures; however, precautions must be taken to prevent the slow growing methanogenic bacteria from being washed out of the system.

b) pH, alkalinity, and volatile acids

The pH within the anaerobic digester is also an important parameter that must be monitored carefully. Many researchers have determined that the allowable pH range for anaerobic systems is from pH 6.0 to 8.0. McCarty [33] stated that the preferred pH is from 7.0 to 7.2, while Dague [7] recommended a pH of 6.8 to 7.2. Parkin and Owen, [44] stated that the accepted range for process efficiency is 6.5 to 7.6. The optimum pH recommended by most researchers is a neutral pH of 7.0 [5,7,14,33,44,61].

The pH tolerance of an organism is usually considered to be a direct reflection of the pH-activity characteristics of that organism's enzymes [5]. Generally, pH effects are caused by changes in the pH which cause the ionization state of various system components to change. For enzymes, the changes in ionization state may occur in either the free enzyme, the enzyme-substrate complex, or the substrate [54]. If the system is affected to the point where the pH falls out of the "preferred" range the system will still operate but an imbalance between the two biochemical stages results, causing acid accumulation within the digester [61]. This imbalance can be caused by changes in organic or hydraulic characteristics, a temperature change, or introduction of toxic substances [44]. The imbalance between the two stages
causes acid accumulation to occur when the acetogenic bacteria produce volatile acids at a faster rate than the methane bacteria are able to decompose them. The digester must have a sufficient buffer capacity, otherwise the imbalance will cause the pH to drop to the point of total reactor failure.

The role of alkalinity is to neutralize the acids that are formed during digestion. Maintaining a sufficient level of alkalinity within the system ensures that slight system imbalances can be handled. McCarty [33] suggested a bicarbonate alkalinity of 2,500 to 5,000 mg/L. This buffer capacity will allow the digester to handle increases in volatile acids with only a minimum drop in the pH [33].

The concentration of carboxylic acids within a smoothly operating system are generally very low, < 100 mg/L [60]. In a stable digester acetic acid is the principal carboxylic acid, but as the digester becomes stressed the concentrations of butyric and propionic acid increase [60]. Volatile acid concentrations above 2,000 to 6,000 mg/L were thought to be toxic to methane organisms, independent of pH [36]. However, McCarty and McKinney [36] tested this theory and found some quite different results. They found that under stressed reactor conditions, as the acids accumulate, the pH drops and the hydrogen ion concentration increases. The hydrogen ions are very toxic to biological systems but can be removed by the addition of alkaline materials, usually lime or sodium hydroxide. What is actually occurring is that one cation is being replaced by another. If the replacement cation is also toxic then conditions will not improve even at a higher pH [36]. Various sodium salts were tested on a series of digesters. The results indicated that the cation concentration was the inhibiting factor and not the anion concentration. This means that the inhibition is based on salt toxicity not volatile acid toxicity. Based on this concept, McCarty and McKinney concluded that relatively high volatile acid concentrations can be tolerated provided they are neutralized with alkaline materials containing a cation of low toxicity.
c) Toxic substances

There are many substances that can be inhibitory to the anaerobic digestion process if they are present in sufficient amounts. The substances which are most commonly reported as inhibitory to anaerobic digestion include inorganics such as alkali and alkaline-earth metals, ammonia nitrogen, sulfide, heavy metals, and a wide variety of organic compounds [7,34,44,54]. In a 1986 publication, Parkin and Owen [44] provided a comprehensive summary of various toxic substances which are shown in Tables 5 and 6, respectively.

Generally, the salts such as sodium, potassium, magnesium, and calcium are beneficial to the digestion process at lower concentrations. In a typical domestic waste stream these salts would not reach inhibitory concentrations. Care must be taken when treating an industrial waste stream because they frequently contain strongly inhibitory concentrations of these salts [35]. One method that is commonly used to reduce the toxic effects is to introduce an ion that is antagonistic to the toxic substance [34]. An example of this would be to introduce potassium to a waste that has toxic levels of sodium.

Ammonia is usually formed in anaerobic treatment from the degradation of wastes containing proteins or urea [34,44]. Ammonia concentrations within the digester are dramatically dependent on pH and temperature [16,34,38,44].

Research has demonstrated that lower concentrations of ammonia are beneficial to the digestion process. Ammonia can be present either in the form of the ammonium ion (NH$_4^+$) or as dissolved ammonia gas (NH$_3$) as is shown by the following equilibrium equation [35,44].

$$\text{NH}_3 + \text{H}_2\text{O} \leftrightarrow \text{NH}_4^+ + \text{OH}^-$$

The control of the pH is very significant in the type of ammonia product that is produced. If the pH is maintained near 7.0 the reactor kinetics favor the NH$_4^+$. An increased pH favors the ammonia gas which is toxic at much lower concentrations than the ammonium
Table 5. Concentrations\(^a\) of various inorganics reported to be inhibitory to anaerobic digestion [44]

<table>
<thead>
<tr>
<th>Substance</th>
<th>Moderately inhibitory</th>
<th>Strongly inhibitory</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium (Na(^+))</td>
<td>3,500 - 5,500</td>
<td>8,000</td>
</tr>
<tr>
<td>Potassium (K(^+))</td>
<td>2,500 - 4,500</td>
<td>12,000</td>
</tr>
<tr>
<td>Calcium (Ca(^{2+}))</td>
<td>2,500 - 4,500</td>
<td>8,000</td>
</tr>
<tr>
<td>Magnesium (Mg(^{2+}))</td>
<td>1,000 - 1,500</td>
<td>3,000</td>
</tr>
<tr>
<td>Ammonia-nitrogen</td>
<td>1,500 - 3,000</td>
<td>3,000</td>
</tr>
<tr>
<td>Sulfide</td>
<td>200</td>
<td>200</td>
</tr>
<tr>
<td>Copper (Cu)</td>
<td>----</td>
<td>0.5 (soluble)</td>
</tr>
<tr>
<td>Chromium VI (Cr)</td>
<td>----</td>
<td>3.0 (soluble)</td>
</tr>
<tr>
<td>Chromium III</td>
<td>----</td>
<td>200 - 260 (total)</td>
</tr>
<tr>
<td>Nickel (Ni)</td>
<td>----</td>
<td>2.0 (soluble)</td>
</tr>
<tr>
<td>Zinc (Zn)</td>
<td>----</td>
<td>1.0 (soluble)</td>
</tr>
</tbody>
</table>

\(^a\)All concentrations in mg/L
Table 6. Concentrations\textsuperscript{a} of various organics reported to be inhibitory to anaerobic digestion [44]

<table>
<thead>
<tr>
<th>Organic</th>
<th>Inhibitory concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formaldehyde</td>
<td>50 - 200</td>
</tr>
<tr>
<td>Chloroform</td>
<td>0.5</td>
</tr>
<tr>
<td>Ethyl Benzene</td>
<td>200 - 1,000</td>
</tr>
<tr>
<td>Ethylene Dichloride</td>
<td>5.0</td>
</tr>
<tr>
<td>Kerosene</td>
<td>500</td>
</tr>
<tr>
<td>Linear ABS (detergent)</td>
<td>1% of dry solids</td>
</tr>
</tbody>
</table>

\textsuperscript{a}All concentrations in mg/L

It is believed that free-ammonia (NH\textsubscript{3}-N) concentrations are the key to controlling ammonia inhibition. Many researchers feel that free-ammonia concentrations greater than 100 mg/L may cause severe toxicity [44]. However, more recent research has suggested that with acclimation, ammonia nitrogen concentrations as high as 8,000 to 9,000 mg/L could be handled successfully by the digester.

Sulfides in anaerobic treatment can result from [34]:

1. Introduction of sulfides with the raw waste.

2. Biological production in the digester from reduction of sulfates and other sulfur containing inorganic compounds.

In an anaerobic environment, sulfides may exist in either soluble, insoluble, or in gaseous (H\textsubscript{2}S) form. The pH within the digester controls the distribution of the produced
sulfides. When insoluble heavy metal sulfides are precipitated they become relatively harmless to the system. It is the soluble sulfides that must be monitored very carefully. The accepted limit for soluble sulfide concentrations is 50 to 100 mg/L. However, tests have shown that with time for acclimation soluble sulfide concentrations of 200 mg/L can be treated with no inhibitory effects [34].

Heavy metals may exist in an anaerobic digester in either soluble or insoluble form. The soluble heavy metal concentrations shown in Table 5 are some of the most common inhibitory metals to anaerobic treatment. It takes only small amounts of these metals to cause serious digester problems. The metals affect the digestion process through inactivating enzymes by reacting with their sulphydryl group [52]. It is believed that the free metal ions are the direct inhibitors in anaerobic treatment [41].

There have been two ways demonstrated to remove significant amounts of heavy metal ions from solution to prevent digester inhibition. The methods are [41] 1) interaction of the metal with the hydrogen sulphide/sulfide systems and 2) interaction with the carbon dioxide/carbonate system. The most commonly used method is the addition of sulfide to create harmless inorganic precipitates. Lawrence and McCarty [27] demonstrated that the use of sulfide could control the toxic effects from the heavy metals copper, zinc, nickel, and iron. Relatively high concentrations of heavy metals can be tolerated if there is a sufficient amount of sulfides present [34]. However, care must be taken because sulfides can also be quite toxic to the anaerobic digestion process.

There are also many organic materials that can be toxic to anaerobic digestion at certain concentrations. Table 6 identifies the inhibitory levels of a few of these various organics.

Continuous feeding has been suggested as a preferred treatment method for wastes high in organic content [34]. By continuously feeding, the digester is able to degrade the
organics as rapidly as they are added which keeps the actual reactor concentrations relatively low [34].

All of the substances that have been discussed can become inhibitory to anaerobic digestion if their concentrations become too high. In most cases, if proper care is taken during system start up, toxic effects can be eliminated by allowing adequate acclimation time for the microbial population within the digester.

2. Operational Elements

a) Solids retention time

The solids retention time (SRT) maintained within a anaerobic digester is one of the key operational elements that must be closely monitored to ensure that microbial washout does not occur. When steady state conditions are maintained within the digester, the SRT is defined as the following equation [6].

\[
\text{SRT (days)} = \frac{\text{mass of solids in system}}{\text{mass of solids removed per day}}
\]

In order for successful anaerobic treatment to occur, the SRT within the digester must be longer than the regeneration time of the slowest growing microorganisms. In anaerobic treatment the slowest reproducing microbes are the methanogens. As previously discussed, the methanogens are the only microbes within the anaerobic treatment process capable of fully stabilizing waste. If the SRT is too short and the methanogens are washed out of the system, acid build-up will occur and the digester will quickly fail.

Many anaerobic treatment systems are designed to operate at a temperature of 35° C. At this temperature the accepted minimum SRT is approximately 10 days [9]. The minimum required SRT within a digester is extremely temperature dependent. The "Q_{10} Rule" states that the biological reaction rates approximately double for every 10° C rise in temperature.
Therefore, detention times of 20 days at 25°C, 10 days at 35°C, and 5 days at 45°C are recommended [46].

It is possible to obtain the desired degree of waste stabilization at lower temperatures by increasing the solids retention time [45]. If the increased SRT provides proper compensation for the decrease in the rate constants, then the desired treatment can be obtained.

b) Organic loading rate

The organic loading rate (OLR) represents the rate at which the substrate enters the treatment system. The OLR affects many different system parameters.

At any given organic loading rate the maximum amount of methane that may be produced is constant. Pfeffer et al [45] demonstrated that the destruction of volatile solids can produce a fixed amount of methane per unit of solids destroyed regardless of the amount of solids destroyed as long as the composition of the solids remains constant. Methane production increases as the SRT increases because of the increased microbial contact time with the substrate.

c) Mixing

Adequate mixing within an anaerobic digester provides a uniform reactor environment. Proper mixing helps disperse metabolic end products and any toxic materials that may be present [44]. It also helps maintain intimate contact between the bacteria, bacterial enzymes, and their substrate [44].

Dague et al [9] reported that intermittent mixing provided increased gas production and increased COD and solid removal compared to continuous mixing. From these experiments, Dague et al [9] concluded that intermittent mixing improves bio-flocculation while continuous mixing results in poor bio-flocculation and inefficient solids separation.
All of these operational elements are very important to the anaerobic digestion process and should be designed carefully to ensure that the desired treatment objectives are achieved.

C. Fundamentals of the Anaerobic Sequencing Batch Reactor

The theory supporting the development of the anaerobic sequencing batch reactor (ASBR) came from the research of the anaerobic activated sludge process by Dague et al [8] in 1966. The ASBR was first studied in laboratory scale experiments by Habben [15] in 1991.

The ASBR process utilizes four phases and is based on batch kinetics. The four phases of the ASBR process are shown in Figure 2. Phase one is known as the feed phase. During this phase the waste stream enters the reactor and is mixed with the biomass. In phase two, which is the react phase, intimate contact of the microorganisms and the waste is achieved with either continuous or intermittent mixing. During this phase the microbial population is able to stabilize the waste by converting it to methane. In phase three the mixing is stopped and the biomass is allowed to settle. During the settling phase the ASBR works as a clarifier eliminating the need for external clarification. Successful clarification is possible because of minimal internal gasification during settling within the ASBR. Minimal internal gasification is achieved because the design of the ASBR utilizes Monod kinetics. Figure 3 illustrates the principal of Monod Kinetics and how they are utilized in the ASBR process. In their anaerobic activated sludge design Dague et al [8] concluded that the settlability of the sludge within a reactor is dependent on the food to microorganism (F/M) ratio within the system. Monod kinetics state that at the start of a reactor cycle the high F/M ratio causes the greatest gas production. As the cycle progresses, the food is utilized and the F/M ratio decreases. By the end of the cycle both the F/M ratio and the gas production are extremely low allowing for excellent settling. Because of these kinetic principles the ASBR achieves lower F/M ratios than the commonly used anaerobic contact process. By achieving lower
Figure 2. The four phases of the ASBR process
Figure 3. F/M variation in the ASBR process
F/M ratios the ASBR is able to avoid the gasification problems commonly associated with the anaerobic contact process.

Phase four occurs after the sludge is settled and consists of decanting the supernatant from the reactor. The reactor then enters the feed phase and continues in a cyclic manner.

The excellent settling characteristics of the biomass within the ASBR allow for relatively long SRT's to be achieved. Habben [15] demonstrated that the ASBR was able to maintain long solid retention times at hydraulic retention times as low as 12 hours while feeding a substrate of nonfat dry milk.

Research by Pidaparti [46] and Schmit [52] illustrated that the ASBR can successfully adapt to lower temperatures. They were able to treat swine waste at temperatures of 35° C, 25° C, and 20° C. At lower temperatures the SRT within the ASBR was actually found to increase due to the lower endogenous decay rate of the microorganisms and the ability of the ASBR to maintain solids.

Research on the ASBR was also conducted at higher temperatures. Kaiser [24] investigated the ability of the ASBR to treat a synthetic waste consisting of nonfat dry milk at thermophilic temperatures. Kaiser found that the ASBR is quite capable of operating at thermophilic temperatures. However, the high rate of endogenous decay created some problems in retaining sufficiently long solids retention times.

D. Leachate Characteristics

Leachate is generated as water infiltrates into the refuse layers within the sanitary landfill. The two factors used to characterize the leachate produced at a particular landfill are the volumetric flow rate at which the leachate is produced and the chemical composition of the leachate.
1. Leachate Quantity

The quantity of leachate generated from a sanitary landfill is highly variable and depends on the design of the landfill site and methods of operation and management of the system [1]. The landfill design is able to control the volume of leachate that is produced by diverting rainfall from entering the landfill site. Landfills are usually designed to control water inputs by means of waterproof covers or by growing suitable plants on the soil covering the waste [28]. After the rainwater is diverted from the refuse emplacement area, it flows into a drainage system which discharges the water into a storm sewer or releases it back into the environment. By allowing less infiltration of water into the fill, there will be a smaller volume of leachate to treat and the risk of leachate contamination is greatly reduced. Before limiting the amount of water that is allowed to enter a landfill, the benefits of a lower leachate volume must be weighed against the disadvantages of a reduction in the rate of decomposition of the wastes within the fill [28,39,55,59].

The nature of the wastes that are placed into the landfill also affects the volume of leachate that is produced. A waste with a high moisture content will enhance the production of leachate within the landfill. Also, the degree of compaction of the waste has an impact on leachate production. Leachate production is generally greater whenever the waste is less compacted, since compaction reduces the filtration rate [28].

All of these factors combine to make it difficult to predict the actual amount of leachate that will be produced at a specific landfill site. The U.S. Army Corps of Engineers has developed the HELP model (Hydrologic Evaluation of Landfill Performance) as an aid to the rapid and economical estimation of the amounts of surface runoff, subsurface drainage, and leachate that may be expected to result from the operation of a wide variety of possible landfill designs [28].
2. Leachate Quality

The chemical composition of the leachate that is generated at a sanitary landfill is also site specific. The composition of leachate may depend on such factors as the fill material (organic content, degradability, solubility), geological conditions, age of the fill, waste composition, etc. [3]. Generally, the composition of leachates is defined in terms of their organic, inorganic, and heavy metal constituents [49]. Although all of these factors affect the leachate composition, the fact that all organic materials in the waste undergo partial or total anaerobic decomposition means that all leachates contain intermediate products of this process [28]. The leachates also tend to contain high concentrations of chemically-reduced inorganic substances, such as ammonia, iron (II) and manganese (II) compounds, and sometimes zinc [50].

Due to the highly variable nature of leachates it is impossible to predict with any accuracy the relative strength of the leachate from any one site. However, it is known that as a landfill matures the composition of leachates change. Landfills proceed through a series of five stabilization phases before final maturation is achieved. These five phases are described in Table 7. All of the events described in Table 7 are encountered at one time or another throughout the life of the landfill provided that the microbial population receives appropriate amounts of moisture and nutrients and they are being inhibited by the presence of toxic material [47]. A landfill never has one "age," but rather a family of different ages associated with the various cells within the landfill complex and their respective progress toward stabilization [47]. In young landfills, as Table 7 indicates, the leachates tend to contain high concentrations of dissolved organic substances, most of which are short-chain volatile acids such as acetic, butyric, and propionic acid [50]. As the landfill ages, the majority of the hydrolyzable organic matter has been fermented, so the organic matter being leached out comes only from the new waste, which as the landfill grows constitutes a
Table 7. Five phases of landfill stabilization [47]

Phase I: Initial Adjustment

- Initial waste placement and preliminary moisture accumulates.
- Initial subsidence and closure of each landfill area.
- Changes in environmental parameters are first detected to reflect the onset of stabilization processes which are trending in a logical fashion.

Phase II: Transition

- Field capacity is approached and leachate is formed.
- A transition from initial aerobic to facultative and anaerobic microbial stabilization occurs.
- The primary electron acceptor shifts from oxygen to nitrates and sulfates with the displacement of oxygen and carbon dioxide in the gas.
- A trend toward reducing conditions is established.
- Measurable intermediates such as the volatile organic fatty acids first appear in the leachate.

Phase III: Acid Formation

- Intermediary volatile organic fatty acids become predominate with the continuing hydrolysis and fermentation of waste and leachate constituents.
- A precipitous decrease in pH occurs with a concomitant mobilization and possible complexation of metal species.
- Nutrients such as nitrogen and phosphorus are released and utilized in support of the growth of biomass commensurate with the prevailing substrate conversion rates.
- Hydrogen may be detected and affect the nature and type of intermediary products being formed.

Phase IV: Methane Fermentation

- The pH returns from a buffer level controlled by the volatile organic fatty acids to one characteristic of the bicarbonate buffering system.
- Oxidation-reduction potentials are at their lowest values, sulfates and nitrates have been reduced to sulfides and ammonia.
- Complexation and precipitation of metal species with sulfides and organic ligands proceed.
Table 7. (continued)

- Leachate organic strength is dramatically decreased in correspondence with increases in gas production.

Phase V: Final Maturation

- Relative dormancy following active biological stabilization of the readily available organic constituents in the waste and leachate.
- Nutrients may become limiting.
- Natural environmental conditions become reinstated.
- Oxygen and oxidized species may slowly reappear with a corresponding increase in oxidation-reduction potential.

Majority of the hydrolyzable organic matter has been fermented, so the organic matter being leached out comes only from the new waste, which as the landfill grows constitutes a progressively smaller fraction of the fill [28]. Table 8 shows the extreme variability of leachate constituent concentrations and how they compare with the leachate from the Iowa City, Iowa landfill that was used throughout this research. The characteristics of landfill leachate vary significantly from site to site and it must be stressed that a successful treatment method for one leachate may not be the best treatment method for the leachate from another landfill.

E. Anaerobic Treatment of Landfill Leachate

The stench, high organic matter content, and volume make the immediate treatment of landfill leachates imperative [28]. Landfill leachates are generally well suited for anaerobic treatment due to the substantial amount of volatile fatty acids found in them. It is these readily degradable acids that account for the bulk of the chemical oxygen demand of many leachates which make them amenable to anaerobic treatment [25]. However, the seasonal variability in volume and chemical composition, make selecting any one treatment method
Table 8. Ranges of leachate constituent concentrations\textsuperscript{a} [49]

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Typical range</th>
<th>Iowa City, Iowa range</th>
</tr>
</thead>
<tbody>
<tr>
<td>BOD\textsubscript{5}</td>
<td>4 - 57,700</td>
<td>275 - 2,850</td>
</tr>
<tr>
<td>COD</td>
<td>31 - 89,520</td>
<td>1,120 - 3,520</td>
</tr>
<tr>
<td>Ammonia-nitrogen</td>
<td>0 - 1,966</td>
<td>15.6 - 109</td>
</tr>
<tr>
<td>Total suspended solids</td>
<td>----</td>
<td>50 - 1,600</td>
</tr>
<tr>
<td>Total volatile suspended solids</td>
<td>----</td>
<td>4 - 630</td>
</tr>
<tr>
<td>pH</td>
<td>3.7 - 8.8</td>
<td>6.1 - 6.6</td>
</tr>
<tr>
<td>Magnesium (Mg\textsuperscript{2+})</td>
<td>17 - 15,600</td>
<td>1.5 - 260</td>
</tr>
<tr>
<td>Copper (Cu)</td>
<td>0.005 - 9.9</td>
<td>0.01 - 0.03</td>
</tr>
<tr>
<td>Chromium (total) 0.2 - 18</td>
<td>&lt;0.02</td>
<td></td>
</tr>
<tr>
<td>Iron (Fe)</td>
<td>4.0 - 2,820</td>
<td>160 - 420</td>
</tr>
<tr>
<td>Zinc (Zn)</td>
<td>0.6 - 370</td>
<td>0.2 - 0.32</td>
</tr>
</tbody>
</table>

\textsuperscript{a}All concentrations except pH in mg/L
anaerobic methods for the treatment of landfill leachate. This section does not attempt to review all of the studies that have been conducted, rather, it focuses on a few selected studies to show the variety of treatment methods that have been analyzed for the treatment of landfill leachate.

The first studies using anaerobic methods for the treatment of landfill leachate were conducted by Boyle and Ham [1] in 1974. Their experiments, which utilized a bench-scale anaerobic digester, demonstrated that BOD reductions of greater than 90% could be achieved for hydraulic retention times greater than 10 days at temperatures in the range of 23 to 30° C. Boyle and Ham were also able to show that aerobic polishing of the anaerobic effluent produced BOD values commensurate with surface water discharge [1].

In 1986, Thirumurthi et al [59] conducted an extensive study using two anaerobic fixed film reactors (AFFR) for the treatment of a high-strength leachate (23,000 mg COD/L, 17,500 mg BOD/L, 1,000 mg Fe/L, and 80 mg Zn/L). System one consisted of an AFFR, the effluent from which was further treated by an aerated lagoon and a settling lagoon in parallel. System two also consisted of an AFFR, but it was not followed by any other treatment. Both AFFRs were operated in the upflow mode and maintained at a temperature of 32° C throughout the study. The leachate for both systems was pretreated to raise the pH and to remove the potentially toxic Zn concentrations.

The organic loading rates that were maintained on each AFFR system were 2.0 and 1.6 kg COD/day/m^3 for system one and two respectively. Because the goal of this study was to achieve a high quality effluent, the organic loading rates were slightly lower than the normal range (2 to 8 kg COD/day/m^3) of volumetric organic loads used in several studies.

Both systems were able to achieve significant reductions in both organic content and metal concentrations. System Ia, which included the aerated lagoon, resulted in greater than 99% removal in BOD, COD, Fe, and Zn. However, the long hydraulic retention time (70
days) in the aerated lagoon resulted in very high concentrations of nitrates and nitrites (138 mg/L) in the effluent. System Ib, which utilized a settling lagoon with the AFFR, resulted in removals greater than 98% for BOD, COD, Fe, and Zn. This system had no nitrification problems, but it produced an effluent of 37.5 mg/L of ammonia-N and 49 mg/L of Kjeldahl-N. System II, which consisted of only an AFFR achieved greater than 97% removal of BOD, COD, Fe, and Zn. Thirumurthi et al concluded that for this high strength leachate the best treatment alternative would utilize a combination of systems Ia and Ib, so that an effluent of low nitrate and ammonia concentrations can be achieved.

In 1983 Henry et al [18] did a pilot study using an anaerobic filter for the treatment of low strength (1,500 to 2,000 mg COD/L) landfill leachate. This was one of the first attempts to treat leachate with an anaerobic filter at an ambient temperature of 25° C +/- 5° C. The experimental set-up consisted of four anaerobic filters of equal volume (0.024 m³). Two of the filters utilized rock media while the other two reactors used plastic biorings. One reactor of each type of filter media was operated in the upflow mode while the two remaining filters were operated as downflow units. All of the filters followed an identical treatment regime. The filters began operation at a hydraulic retention time of 72 hours, which corresponded to a loading rate of approximately 0.62 kg COD/m³/day. After an initial acclimation period the hydraulic retention times were reduced to 24 hours and eventually to 12 hours. At the 12 hour HRT the loading rate on the filters was approximately 3.34 kg COD/m³/day. Throughout this study all four filters achieved similar removal rates. The effluent COD values for all the filters varied between 200 to 540 mg/L which corresponded to removal rates of 67 to 86%. Henry et al concluded that the anaerobic filter can provide a simple an effective method of treatment for treating low strength leachates.

A 1988 study by Kennedy et al [26] evaluated the feasibility of using the upflow blanket filter (UBF) and the downflow stationary film (DSF) reactor for the anaerobic
treatment of a high strength (19,560 mg COD/L) landfill leachate. The leachate used in this study was pretreated with a lime solution in order to precipitate heavy metals that might inhibit anaerobic bacteria. The DSF reactor was filled with needle punched polyester support material. The ratio of packing material surface area to reactor volume was 75 m²/m³ [26]. The DSF was continuously fed at the top of the reactor with the effluent being collected from the bottom of the reactor. The ratio of recirculation to feed for this system was 4 to 1. The UBF reactor was identical in size to the DSF reactor. However, the UBF reactor had plastic biorings filling the top third of the reactor volume. This reactor also had a recirculation to feed ratio of 4 to 1, but this reactor was continuously fed from the bottom.

After an initial start-up period, both reactors were operated at three different steady states at HRTs between 4.2 and 1.5 days. The organic loading rates that were applied to these systems varied from 4.8 to 14.7 kg COD/m³/day. Both of these systems performed well at all of the loading rates, with the UBF achieving TCOD removals ranging from 95.8 to 96.3% and the DSF achieving removals from 94.0 to 96.8%. The DSF reactor effluent consistently had higher concentrations of TSS, VSS, and FSS than the UBF system. However, this was expected due to the downflow operation of the DSF. Kennedy et al [26] concluded that both the DSF and UBF reactors can successfully treat this leachate while maintaining an organic loading rate of 14.5 kg COD/m³/day and an HRT of 1.5 days. The major problem encountered during this study involved the pretreatment of the leachate with the lime solution. This pretreatment resulted in the production of precipitates that eventually caused clogging and pumping problems within both systems. Therefore, it was concluded that the lime solution should not be used in future testing of this system.

All of these systems represent various methods that have been studied for the anaerobic treatment of landfill leachate. Each treatment method has its advantages and
disadvantages, and due to tremendous variability of leachate from site to site, the selection of a treatment method for a particular leachate must be made with great care and consideration.
III. EXPERIMENTAL SETUP

A. Phase One Reactor Configuration

Phase One was conducted using two identical 14-liter Plexiglas reactors. Figures 4 and 5 show a typical ASBR reactor and a schematic of the entire ASBR system, respectively.

Both reactors were constructed of 0.5-in thick Plexiglas and were cylindrical in shape. The reactors were 36 in long with an inside diameter of 5 in and each was fitted with a 9-in diameter flange on both the top and bottom. 9-in diameter plates constructed of 0.5-in thick Plexiglas were fitted to the flanges with twelve equally spaced 3/8-in diameter bolts, which were secured with nuts and washers. Circular grooves were cut into both the plates and the flanges to allow for the placement of an O-ring, which was used to seal the reactor. This construction yielded a reactor with a total volume of 14 liters. A working liquid volume of 12 liters was used.

Each reactor was fitted with nine effluent ports. They were equally spaced 4 in apart along the length of the reactor with the first port 2 in from the top of the reactor. Each port had the following characteristics; an inside diameter of 3/8 in, outside diameter of 5/8 in, and a length of 1 in. The ports were fitted with Tygon tubing, clamped off, and were used as needed throughout the research.

The plate on the top of the reactor was fitted with three ports. Two of these ports had an inside diameter of 3/8 in and an outside diameter of 5/8 in, each of which were 1 in long. Both ports allowed for the removal of biogas and foam from the reactor. However, the main purpose of the second port was to allow for the return of foam into the reactor. The third port was fitted with a 0.5 in diameter stainless steel rod. This rod was fitted into the reactor with a Swagelok fitting and extended the length of the reactor. The bottom of the rod was fitted with a copper ring diffuser. This rod and diffuser combination was used for
Figure 4. ASBR reactor configuration
Figure 5. Schematic of ASBR system
biogas mixing of the reactor contents.

All of the biogas and foam produced within the reactor was transported from the reactor through the top ports and entered the gas-foam separation bottle. The reactor and gas-foam separator were connected with Tygon tubing. Figure 6 shows the gas-foam separation apparatus. This apparatus was needed to keep foam and other particles from entering the recirculation system and clogging the diffuser. The gas-foam separator consisted of a 4-liter aspirator bottle which was equipped with a discharge port at the bottom. The bottle was sealed with a number 10 rubber stopper. The stopper had three holes drilled through it, each of which was fitted with a 3/8-in piece of glass tubing. Two of the glass tubes were approximately 2 in long. The third piece of glass tubing was approximately 10 in long. The gas and foam entered the separator through the long glass tube. After entering the aspirator bottle the gas was separated from the foam and exited the aspirator bottle through the two smaller tubes. One of the tubes sent the biogas to the recirculation pump and the other tube sent biogas to the gas measurement system. The foam was returned to the reactor through the discharge port located at the bottom of the aspirator bottle. This discharge port was connected to one of the exit ports located on the top of the reactor with Tygon tubing.

The gas recirculation system consisted of Cole Parmer variable (6-600) rpm speed pump and a Masterflex solid state speed controller. The pump was fitted with a size 18 pump head which held 8-mm inside diameter Masterflex neoprene hose. This pump recirculated the biogas through the stainless steel tube and out of the copper ring diffuser. The diffuser was constructed of 0.5-in diameter copper tubing and had an outside diameter of 4 in. The copper ring had 8 equally spaced 1/16-in diameter holes drilled into the top of it. These holes allowed the recirculated biogas to thoroughly mix the reactor contents. The gas recirculation pump was controlled by a 4-outlet 10-program Chronotrol timer. This enabled the mixing times to be easily changed.
Figure 6. Gas-Foam separation bottle
The gas bag, which is shown in Figure 5, served as a biogas reservoir that allowed gas to fill the reactor head space that was formed during effluent decanting. The gas bag consisted of a beach ball which had the inlet valve removed. This was replaced with a 3-way fitting which allowed gas to flow in or out of the gas bag.

All gas that was produced passed through the gas bag and into an observation bottle and hydrogen sulfide scrubber (Figure 7). Both the observation bottle and the scrubber consisted of 1-liter glass bottles which were sealed with rubber stoppers. Each stopper was fitted with two pieces of 3/8-in outside diameter glass tubing. The lengths of the glass tubes in each bottle were 5 in and 1 in, respectively. The purpose of the observation bottle, which was half filled with water, was to give a visual indication of gas production. The gas entered the observation bottle through the long glass tube, bubbled through the water, and escaped out the shorter tube. The gas then entered the H2S scrubber, which was filled with steel wool, through the other long glass tube. As the gas passed through the scrubber bottle the steel wool removed the hydrogen sulfide from the biogas. By removing the hydrogen sulfide from the biogas, the scrubber was able to prevent damage to the gas meters.

After the gas passed through the scrubber bottle it exited the small glass tube and was transported through a gas sampling port. The gas sampling port was constructed of a glass tube that was 2.5 in long and had an outside diameter of 0.5 in. This tube was fitted with 3/8-in glass tubing on both ends to allow for connection into the entire system. A 5/16-in piece of glass tubing was installed into the center of the sampling port. This was fitted with a rubber septum. The septum allowed gas samples to be drawn without any contamination from the outside air occurring. The gas then passed through a wet tip gas meter (Figure 8). These gas meters were manufactured by the Rebel Wet Tip Gas Meter Company.

The leachate feeding system consisted of a Masterflex peristaltic pump with speed controller. The pump was fitted with a size 18 pump head which held 8-mm inside diameter
Figure 7. Observation bottle and hydrogen sulfide scrubber
Figure 8. Typical wet tip gas meter
Masterflex neoprene hose. This pump fed the leachate into the reactor through the lowest side port as shown in Figure 5.

The effluent decant system consisted of a Cole Parmer variable (1-100) rpm speed pump and a Masterflex solid state speed controller. The pump was fitted with a size 18 pump head which held 8-mm inside diameter Masterflex neoprene hose. The decanting was done from various side ports depending upon the current operating conditions of the system. Both the feed and decant pumps were controlled by the Chronotrol timer previously described.

B. Phase Two Reactor Configuration

In Phase Two an aerobic reactor was used in series with the ASBR system. This section will be used to describe the configuration of the aerobic reactor. The aerobic reactor was constructed of 1/4 in thick Plexiglas. Figure 9 shows a schematic of the aerobic system used for this research. The volume of this reactor was 11.5 liters with a working liquid volume 8 liters.

The reactor was rectangular in shape with two walls 5 in by 24 in and the other two walls 6 in by 24 in. An 8-in long piece was inserted in the back of the reactor and angled toward the reactor bottom. The purpose of this piece was to force the biomass to the front of the reactor where the diffuser was located.

The diffuser was inserted through a hole drilled in the front of the reactor. It consisted of a 1/4-in stainless steel tube that extended for 5 in along the bottom of the reactor. The top of this piece had eight equally spaced 1/16-in diameter holes drilled in it. A portion of the stainless steel tubing protruded from the reactor. This was fitted with Tygon tubing that was connected to the mixing pump. The mixing apparatus consisted of a Cole Parmer peristaltic pump with speed controller. This pump was fitted with a size 18 pump head which held 8-mm inside diameter Masterflex neoprene hose. One end of the neoprene hose was connected to
Figure 9. Schematic of aerobic system
the diffuser with Tygon tubing while the other end of the neoprene hose was connected to the
diffuser with Tygon tubing while the other end of the neoprene hose was open to the
atmosphere. This allowed the reactor to be mixed with air.

The feeding system consisted of a Masterflex peristaltic pump with speed controller.
The pump was fitted with a size 16 pump head which held 1/16-in inside diameter Tygon
tubing. This pump fed the reactor through Tygon tubing which was draped over top of the
reactor as shown in Figure 9.

The decant system consisted of a Masterflex 60 rpm constant speed pump. The pump
was fitted with a size 18 pump head which held 8-mm inside diameter Masterflex neoprene
hose. The decanting was done with Tygon tubing in the same manner as the feeding was
conducted. All of the pumps used in this system were controlled by a 4-outlet 10-program
Chronotrol timer.
IV. EXPERIMENTAL PROCEDURES

A. Leachate Preparation

The leachate used in this research was obtained from the Iowa City, Iowa, municipal waste landfill. This landfill is 154 acres in size and has been in operation since 1972. The leachate produced at the landfill enters drainage pipes and is transported to an observation well. From the observation well, the leachate flows into a lift station and on to the Iowa City wastewater treatment plant. The leachate for this project was collected from the observation well approximately every three weeks. The waste was obtained by lowering a submersible pump into the well and drawing the leachate out. The waste was then transported to the lab and was refrigerated at approximately 4° C until needed. Through experimentation it was determined that the addition of supplemental nutrients was not required. However, the pH of the leachate had to be adjusted to ensure that the proper pH of the system would be maintained. It was determined that the addition of 2.75 gm of sodium bicarbonate per liter of leachate would sufficiently raise the pH within the reactor and provide adequate buffering capacity. The leachate was stored and fed in 20-liter carboys, so feed preparation consisted of simply adding 55 gm of sodium bicarbonate to each carboy of leachate prior to feeding.

B. Reactor Operation

1. Phase One

Both of the anaerobic sequencing batch reactors used during phase one were seeded on October 10, 1992. Reactor one was seeded with granular sludge which had been grown and maintained at 35° C in a similar ASBR system using nonfat dry milk as a substrate. Reactor two was seeded with non-granular sludge obtained from the anaerobic digesters at the City of Ames wastewater treatment plant. Both of the ASBR systems used in this
research were operated in a constant temperature room that was maintained at 35°C ± 0.5°C.

Because the granular sludge had been operating with milk as its substrate, the effect of changing to a leachate substrate on the granules was uncertain. Due to the uncertainty surrounding the stability of the granular sludge the transition of substrate from milk to leachate was done gradually. The steps selected for the transition from milk to leachate were based on the results of an anaerobic respirometer test. The respirometer test was able to determine the relative biodegradability of the leachate. The testing procedures used for the respirometer study are discussed in detail in the appropriate section.

After the reactors were seeded, a 40-day start-up period was needed for the sludge to acclimate to the new system. During this time the reactors were fed milk substrate with a COD strength of 3.0 g/L and were operated at an HRT of 48 hours. The load on the reactors during this time was 1.5 g/L/day. The milk substrate was prepared by mixing 45 gm of NFDM, 30 gm of sodium bicarbonate, and 5 ml of trace nutrients with 15 liters of tap water. Tables 9 and 10 show the composition of the NFDM and the trace nutrients, respectively. Once the system was running smoothly the transition from milk to leachate began. The transition was done by mixing raw leachate with the milk substrate. The COD strength of the leachate varied each time it was obtained from the landfill but during the transitional period it was approximately 3.0 g/L. Because the leachate was close to the same strength as the milk the mixture of leachate and milk was done on a volumetric basis. The transition was done in three steps as recommended from the results of the anaerobic respirometer testing. In the first step, a mixture of 2/3 milk and 1/3 leachate was used. This consisted of mixing 10 liters of milk substrate with 5 liters of leachate. The reactors were operated under these conditions for approximately 40 days. During this time all of the various laboratory tests were conducted. These tests are shown in Table 11 and will be
Table 9. Properties of non-fat dried milk, NFDM [20]

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical Oxygen Demand (gm COD/gm NFDM)</td>
<td>1.04</td>
</tr>
<tr>
<td>Five-day Biochemical Oxygen Demand (gm BOD₅/gm NFDM)</td>
<td>0.49</td>
</tr>
<tr>
<td>Total Kjeldahl Nitrogen (%)</td>
<td>5.4</td>
</tr>
<tr>
<td>Total Phosphate as PO₄ (%)</td>
<td>2.2</td>
</tr>
<tr>
<td>Lactose (%)</td>
<td>51.0</td>
</tr>
<tr>
<td>Protein (%)</td>
<td>&gt;36.0</td>
</tr>
<tr>
<td>Fat (%)</td>
<td>&lt;1.0</td>
</tr>
<tr>
<td>Ash (%)</td>
<td>8.2</td>
</tr>
<tr>
<td>Trace Minerals:</td>
<td></td>
</tr>
<tr>
<td>Iron (ppm of NFDM)</td>
<td>4.6</td>
</tr>
<tr>
<td>Nickel (ppm of NFDM)</td>
<td>1.0</td>
</tr>
<tr>
<td>Cobalt (ppm of NFDM)</td>
<td>0.8</td>
</tr>
<tr>
<td>Molybdenum (ppm of NFDM)</td>
<td>3.0</td>
</tr>
<tr>
<td>Zinc (ppm of NFDM)</td>
<td>15.0</td>
</tr>
</tbody>
</table>
Table 10. Recipe for mineral stock solution [20]

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Quantity</th>
<th>Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>FeCl$_2$·4H$_2$O</td>
<td>35.60 gm/L</td>
<td>0.020 gm Fe/gm NFDM</td>
</tr>
<tr>
<td>ZnCl$_2$</td>
<td>2.08 gm/L</td>
<td>0.002 gm Zn/gm NFDM</td>
</tr>
<tr>
<td>NiCl$_2$·6H$_2$O</td>
<td>4.05 gm/L</td>
<td>0.002 gm Ni/gm NFDM</td>
</tr>
<tr>
<td>CoCl$_2$·6H$_2$O</td>
<td>4.04 gm/L</td>
<td>0.002 gm Co/gm NFDM</td>
</tr>
<tr>
<td>MnCl$_2$·4H$_2$O</td>
<td>3.61 gm/L</td>
<td>0.002 gm Mn/gm NFDM</td>
</tr>
</tbody>
</table>

described in detail in the the next section. After 40 days the reactors were operating smoothly. Step two was conducted in the same manner as step one only the feed mixture consisted of 1/3 milk and 2/3 leachate. Again the system was operated for approximately 40 days.

On February 5, 1993 both ASBR systems began treating substrate consisting of 100% leachate. At this time the reactors were still operating at a 48-hr HRT. Initially it was planned to operate the reactors over a variety of loadings at various HRTs. However, this goal became unattainable as summer approached and flood waters overran the entire midwest. The vast amounts of rain water that fell in the Iowa City area caused the strength of the leachate samples to drop dramatically. To combat this problem it was decided to operate the reactors based solely on HRT and let the load fluctuate with the strength of the leachate samples. The HRTs that were to be tested included 48, 36, 24, 18, and 12 hours. The reactors were allowed to achieve a pseudo steady-state before samples were analyzed at the set HRT. Pseudo steady-state was defined by the consistent daily production of methane (+/- 5%). Once steady-state was achieved, a data point was completed by analyzing...
Table 11. Testing parameters and frequency [52]

<table>
<thead>
<tr>
<th>Test Parameter</th>
<th>Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas production</td>
<td>daily</td>
</tr>
<tr>
<td>Gas composition</td>
<td>2/wk</td>
</tr>
<tr>
<td>pH</td>
<td>2/wk</td>
</tr>
<tr>
<td>Alkalinity</td>
<td>at data point</td>
</tr>
<tr>
<td>Volatile fatty acids</td>
<td>at data point</td>
</tr>
<tr>
<td>TCOD removal %</td>
<td>at data point</td>
</tr>
<tr>
<td>Solids removal</td>
<td>at data point</td>
</tr>
</tbody>
</table>

all of the performance parameters of the ASBR system. The performance parameters that were analyzed and their frequency of analysis are shown in Table 11. The tests were conducted three times and averaged for accuracy.

The reactors were operated using four cycles per day. The length of each cycle was 6 hours. The length of each phase within a 6-hr cycle and the mixing frequency is shown below.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed Phase</td>
<td>0.25 hours</td>
</tr>
<tr>
<td>React Phase</td>
<td>4.5 hours</td>
</tr>
<tr>
<td>Settling Phase</td>
<td>1.0 hours</td>
</tr>
<tr>
<td>Decant Phase</td>
<td>0.25 hours</td>
</tr>
<tr>
<td>Mixing</td>
<td>2 min every 0.5 hours</td>
</tr>
</tbody>
</table>

The length of the phases varied slightly as the HRT was lowered because more time was required to feed and decant as the daily volume of leachate being treated increased.
However, these changes were insignificant because variable speed pumps allowed pumping rates to be increased as needed.

Reactor one, which had been seeded with granular sludge, performed well throughout the entire range of HRTs. Problems did arise with reactor two, which had been seeded with non-granular sludge. It never seemed to aclimate well to the leachate. It was operated at a 48-hr HRT for four months and only achieved a TCOD removal of 81.0%. After it was switched to an HRT of 36 hours its performance dramatically declined. Gas production decreased as did the percentage of TCOD removal. The reactor was reseeded but its performance did not improve. It was operated at the 36-hr HRT for four months and was then shut down.

2. Phase Two

The aerobic polishing unit was seeded on August 13, 1993, with sludge obtained from the activated sludge tank at the City of Cedar Rapids wastewater treatment plant. This unit was operated at room temperature (22-24° C).

The aerobic polishing unit operated as a continuously fed, intermitantly decanted reactor. The purpose of this reactor was to determine if an aerobic polishing unit would enable the treatment system to achieve direct discharge requirements. The aerobic reactor, which was operated at a 24-hr HRT, was fed the effluent from the ASBR which was also operating at an HRT of 24 hours. The ASBR operated at a 24-hr HRT because that is a likely HRT for a real world application. The effluent from the ASBR was collected and then transferred to the aerobic unit's feed container. No nutrients or buffers were added to the ASBR effluent before it was fed to the aerobic unit.

The aerobic system was operated using eight cycles per day. The length of each cycle was 3 hours. During each 3-hr cycle the reactor was fed and mixed continuously until the
final 15 minutes, during which the biomass was allowed to settle and one liter of effluent was drawn out of the top of the reactor.

The tests that were conducted on the aerobic unit's effluent included those done on the ASBR except for the biogas testing. However, because direct discharge limits were trying to be attained additional tests were conducted. The aerobic effluent was also tested for the following constituents: carbonaceous biochemical oxygen demand, nitrogeneous biochemical oxygen demand, Phosphorous, Nitrogen, and various metals. These additional tests were conducted by the Analytical Services Laboratory personnel at Iowa State University. Phase two lasted for six weeks and was then shut down.

C. Laboratory Analyses

1. Anaerobic Respirometer Testing

   The anaerobic respirometer testing was conducted prior to the introduction of leachate in the treatment system. This test was performed using the Challenge ANR-100 anaerobic respirometer. The test was initiated by selecting combinations of milk and leachate, each of which had a volume of 30 ml. The milk and leachate solutions were combined with 20 ml of reactor biomass and 635 ml of buffer solution. These constituents were placed into 700 ml testing cells. Tables 12 and 13 show the buffer solution recipe and the contents of each testing cell, respectively. This apparatus was then placed into a 35° C incubator. The respirometer was connected to a computer which recorded the cumulative volume of biogas produced during the experiment. These results, which are discussed in the results section, indicated that the leachate was quite biodegradable.

2. Gas Production

   The amount of biogas produced was recorded by the tipping gas meter previously described. Each day the reading on the gas meter was recorded. By subtracting the previous
Table 12. Buffer solution recipe

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaHCO₃</td>
<td>1,200 mg/L</td>
</tr>
<tr>
<td>KH₂PO₄</td>
<td>270 mg/L</td>
</tr>
<tr>
<td>K₂HPO₄</td>
<td>350 mg/L</td>
</tr>
</tbody>
</table>

Table 13. Contents of each testing cell

<table>
<thead>
<tr>
<th>Component</th>
<th>Cell 1</th>
<th>Cell 2</th>
<th>Cell 3</th>
<th>Cell 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Milk Substrate</td>
<td>30 ml</td>
<td>20 ml</td>
<td>10 ml</td>
<td>-----</td>
</tr>
<tr>
<td>Leachate Substrate</td>
<td>-----</td>
<td>10 ml</td>
<td>20 ml</td>
<td>30 ml</td>
</tr>
<tr>
<td>Buffer Solution</td>
<td>635 ml</td>
<td>635 ml</td>
<td>635 ml</td>
<td>635 ml</td>
</tr>
<tr>
<td>Reactor Biomass</td>
<td>20 ml</td>
<td>20 ml</td>
<td>20 ml</td>
<td>20 ml</td>
</tr>
<tr>
<td>Total Volume</td>
<td>685 ml</td>
<td>685 ml</td>
<td>685 ml</td>
<td>685 ml</td>
</tr>
</tbody>
</table>
days reading from the new reading the volume of biogas produced during the last 24-hr period was obtained. This reading was usually taken at the same time every day because gas production decreases as the cycle progresses due to the decreasing F/M ratio. The barometric pressure and temperature were recorded at the same time the gas reading was taken. The volume of gas that was recorded was then corrected to standard pressure and temperature. Standard pressure and temperature are 760 mm Hg and 273 K respectively. The volume of gas produced at STP was calculated using the following equation:

\[ V_S = \frac{(V_2 - V_1) (P) (T_s)}{(P_s) (T+273)} \]

where:
- \( V_S \) = Cumulative volume of gas produced daily at STP (liters)
- \( V_2 \) = Cumulative volume of gas produced current day (liters)
- \( V_1 \) = Volume of gas produced previous day (liters)
- \( P \) = Daily barometric pressure (mm Hg)
- \( T \) = Daily temperature at gas meter (°C)
- \( T_s \) = Temperature at STP (273 K)
- \( P_s \) = Pressure at STP (760 mm Hg)

3. Gas Composition

The composition of the biogas produced from the ASBR was analyzed twice a week during data collection using gas chromatography (GC). The GC was set to analyze the biogas for \( \text{CH}_4 \), \( \text{CO}_2 \), and \( \text{N}_2 \). The operating conditions and parameters for the GC are listed in Table 14.

A standard gas was used as a basis for the analysis of the biogas. The standard gas consisted of 70% \( \text{CH}_4 \), 25% \( \text{CO}_2 \), and 5% \( \text{N}_2 \) (concentrations +/- 0.5%). This composition
Table 14. Gas chromatograph operating conditions and parameters [52]

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas Chromatograph</td>
<td>Hewlett Packard 5730A</td>
</tr>
<tr>
<td>Column</td>
<td>6 ft x 3 mm I.D. stainless steel</td>
</tr>
<tr>
<td>Packing</td>
<td>Poropak Q, 80/100 mesh</td>
</tr>
<tr>
<td>Temperature</td>
<td>Ambient</td>
</tr>
<tr>
<td>Carrier gas Helium</td>
<td></td>
</tr>
<tr>
<td>Flow rate</td>
<td>30 ml/min</td>
</tr>
<tr>
<td>Column pressure</td>
<td>60 psig</td>
</tr>
<tr>
<td>Detector</td>
<td>Thermal conductivity</td>
</tr>
<tr>
<td>Temperature</td>
<td>150°C</td>
</tr>
<tr>
<td>Bridge current</td>
<td>150 mA</td>
</tr>
<tr>
<td>Sensitivity</td>
<td>10 mA</td>
</tr>
<tr>
<td>Injection point temperature</td>
<td>100 °C</td>
</tr>
<tr>
<td>Sample size</td>
<td>0.9 ml</td>
</tr>
</tbody>
</table>

of the standard gas was chosen because it is a typical biogas composition from anaerobic digesters.

All of the samples were taken with a Hamilton Gas-tight #1001TLL syringe. The volume of all samples used for GC analysis was 0.9 ml. During each biogas analysis three standard gas samples were analyzed for comparison. Samples were analysed in duplicate and averaged for accuracy.

4. pH

The pH of the reactor effluent was monitored twice a week. The pH was tested using an Altex Instruments Model 4500 digital pH meter with a Markson standard glass membrane probe. The pH meter had an accuracy of +/- 0.01 pH units. All samples that were tested for
pH were drawn from the reactor immediately before the test was done. This helped to eliminate loss of CO₂ to the atmosphere prior to testing. If a sample is allowed to sit before testing the carbon dioxide in the sample escapes to the atmosphere causing the pH to increase. Prior to checking the pH, the pH meter was calibrated using two buffer solutions. One buffer had a pH of 7.00 and the other buffer had a pH of 4.00 or 10.00. The second buffer used depended on the estimated pH of the sample.

5. Total Alkalinity

The total alkalinity of the reactor effluent was determined following the procedure outlined in Standard Methods [58]. This test was conducted at each data point and as needed to check the health of the reactors. The test was conducted by obtaining a 25 ml effluent sample. This sample was titrated to a pH of 4.5 using 0.1 N H₂SO₄ acid solution. The total alkalinity was then found using the following equation from Standard Methods [58]:

\[
\text{Total Alkalinity (mg/L as CaCO}_3) = \frac{(50,000) \times (N) \times (\text{ml H}_2\text{SO}_4)}{\text{ml of sample}}
\]

where:
- 50,000 = equivalent weight of CaCO₃ (mg/equivalent)
- N = normality of acid solution (0.1 N)
- ml H₂SO₄ = volume of acid solution used in titration (ml)
- ml sample = volume of sample used in analysis (ml)

6. Total Volatile Acids

The total volatile acids in the reactor effluent were determined following a modified distillation method. This test was conducted at each data point and as needed to check the health of the reactors. The test was performed by distilling a mixture consisting of 100 ml of
sample with 100 ml nanopure water and 5 ml concentrated sulfuric acid. After distillation the sample was titrated to a pH of 8.3 with 0.1 N NaOH. After following the testing procedures the amount of total volatile acids was then calculated using the following equation from Standard Methods [58]:

\[
\text{Total Volatile Acids (mg/L of acetic acid) =} \frac{(60,000) \times (N) \times (\text{ml NaOH})}{(0.7) \times (\text{ml of sample})}
\]

where:

- 60,000 = equivalent weight of acetic acid (mg/equivalent)
- \(N\) = normality of the sodium hydroxide (0.1 N)
- \(\text{ml NaOH}\) = volume of NaOH solution used in titration (ml)
- 0.7 = recovery factor
- \(\text{ml sample}\) = volume of sample used in analysis (ml)

7. Chemical Oxygen Demand

The total chemical oxygen demand (TCOD) was measured on the reactor influent and effluent following the Closed Reflux Titrimetric Method (method #508B) as outlined in Standard Methods [58]. This test was conducted at each data point and as needed to check the health of the reactors. This test measures the oxygen equivalent of organic matter in the sample which can be chemically oxidized with a strong oxidizer [20]. The soluble chemical oxygen demand (SCOD) was also calculated for the reactor influent and effluent. The SCOD test was conducted in the same manner as the TCOD only the samples were filtered prior to testing. The samples were filtered through Fisher Scientific 2.4-mm diameter glass fiber filters with a pore size of 0.45 μm. After filtering the SCOD samples, both TCOD and SCOD tests
are conducted simultaneously. Both tests were run in duplicate and averaged. Once the testing was completed the amount of TCOD and SCOD remaining in the samples was calculated using the following equation from Standard Methods [58]:

\[
\text{COD as mg O}_2/L = \frac{(A - B) \cdot (8,000) \cdot (M)}{\text{ml of sample}}
\]

where:

- \( A \) = volume of FAS titrant used for blank (ml)
- \( B \) = volume of FAS titrant used for sample (ml)
- \( M \) = molarity of FAS titrant
- \( \text{ml sample} \) = volume of sample used in analysis (ml)

8. Solids

The calculation of the amount of solids within the reactors and in the effluent was also a very important testing parameter. The tests for total and volatile suspended solids were performed according to the procedures outlined in Standard Methods [58]. The filters used for this test were Fisher Scientific 9.0-mm diameter glass fiber filters with a pore size of 0.45 \( \mu \text{m} \). Each solids test was performed in triplicate and averaged for accuracy. After the tests were performed, the total and volatile suspended solids in the samples were calculated using the following equations:

\[
\text{TSS (mg/L)} = \frac{(B - A) \cdot (1,000 \text{ mg/gm}) \cdot (1,000 \text{ ml/L})}{\text{ml of sample}}
\]
where:

- \( B \) = weight of filter paper + weighing dish + residue before ignition (gm)
- \( A \) = weight of filter paper + weighing dish (gm)
- \( m_l \text{ sample} \) = volume of sample used in analysis (ml)

\[
VSS (mg/L) = \frac{(B - C) (1,000 \text{ mg/gm}) (1,000 \text{ ml/L})}{m_l \text{ of sample}}
\]

where:

- \( B \) = weight of filter paper + weighing dish + residue before ignition (gm)
- \( C \) = weight of filter paper + weighing dish + residue after ignition (gm)
- \( m_l \text{ sample} \) = volume of sample used in analysis (ml)

9. Automated Image Analysis

The automated image analysis (AIA) was conducted on the reactor contents to determine how the biomass was affected by the introduction of leachate into the system. This test was performed on an adac system 1200 computer using LeMont Scientific, Inc. software. The AIA was conducted by placing a well mixed reactor sample onto a well slide which was then placed under a microscope. The microscope was connected to the computer through a video camera. The computer was able to analyze the biomass particles for a variety of characteristics. The determination of the particle size distribution of the biomass was the primary goal of this test. The results of these tests are discussed in the following section.
V. RESULTS AND DISCUSSION

The following section is a discussion of the performance of the ASBR treating municipal landfill leachate at various hydraulic retention times with and without aerobic polishing.

A. Phase One Results

Due to the tremendous variation in the COD strength of the leachate throughout the duration of this research, it was determined that instead of attempting to hold a constant load on the system, it was more practical to hold the hydraulic retention time at a set rate. Holding the HRT constant allowed the load on the system to vary with the strength of the leachate. There were no adverse effects attributable to the fluctuations in the loading rate during this research.

1. Anaerobic Respirometer Results

The anaerobic respirometer testing was conducted to determine the relative biodegradability of the landfill leachate. The results of this test indicated that by integrating the leachate into the system in three volumetrically equal steps a successful transition could be performed. This test, which was conducted using various combinations of leachate and milk, indicated that the leachate stream was highly degradable (Figure 10). The inhibition occurring in the 66% and 100% leachate samples is attributed to the use of sludge which was unclimatized to the leachate substrate. This can be seen in the early flattening out of the respective lines shown in Figure 10.

The relative success of the anaerobic respirometer testing led to the development of the following transition schedule for the integration of the leachate into the treatment system. The first step was to utilize a feed mixture of 1/3 leachate and 2/3 milk. In step
Figure 10. Anaerobic respirometer testing of various combinations of milk and leachate
two, the mixture was 2/3 leachate and 1/3 milk. The final step in the transitional process was the implementation of a 100% leachate substrate into the treatment system. During the transitional phase, the system was constantly tested to indicate if the leachate was having any adverse effects on the system.

2. Transitional Phase

The transition from the treatment of milk to the treatment of leachate was performed in the three incremental steps previously described. During this transitional period all of the typical reactor performance parameters were monitored to ensure that the health of the system was maintained. Figures 11 and 12 show the variation in TCOD and SCOD removal rates for both reactors during the transition from milk to leachate substrate, respectively. The percentage of COD removal is calculated using the following equation.

\[
\text{Removal, \%} = \frac{(A - B) \times 100}{A}
\]

where:

\begin{align*}
A &= \text{influent COD concentration, mg/L} \\
B &= \text{effluent COD concentration, mg/L}
\end{align*}

Both figures 11 and 12 indicate that a drop in removal rates occurred as each step was implemented. Throughout the transition the non-granular reactor was affected more severely by the change in substrate. Figure 12 shows that COD removal rates for the non-granular reactor dropped quite significantly when it started treating a 100% leachate substrate.
Figure 11. COD removal efficiency for the granular reactor during the transition from milk to leachate
Figure 12. COD removal efficiency for the non-granular reactor during the transition from milk to leachate.
3. Leachate Treatment

a) Hydraulic retention time

The continued variation in the strength of the leachate throughout the study made it impractical to hold the system load at a constant rate. Therefore, it was decided that the best approach would be to hold the HRT constant and let the load on the system vary with the waste strength. Figure 13 shows the variation of the leachate strength for the duration of this research. It must be stressed that during this experiment the order of testing was to start from an HRT of 48 hours and proceed downward until an HRT of 12 hours was reached.

The problems associated with letting the load vary were minimal. The variation of leachate strength never caused the load to suddenly become too strong for the system to handle. Rather, the leachate was continually becoming weaker as the flood waters inundated the landfill area. This created instances when the COD removal rates decreased due to the weak leachate stream. There were also instances when the weakening leachate caused the load to decrease even though the hydraulic retention time was being reduced.

Figures 14 and 15 show how both the load and COD removal rates fluctuated throughout the research for both reactors. The granular reactor (Figure 14), which was operated over 5 different HRTs, was able to maintain fairly consistent COD removal rates, even though the load on the system was varying throughout the experiment. The non-granular reactor (Figure 15) was only operated at two different HRTs, so the performance data for it are presented over time instead of HRT. Figure 15 shows that the non-granular reactor was severely affected by the variations in the loading rate. The variation in the COD load on the system was a function of the leachate strength and the HRT.

b) Biogas production

Monitoring the production of biogas by an anaerobic reactor is the most accurate way to analyze the performance of the system. Figures 16 and 17 show the total biogas and
Figure 13. Fluctuations in leachate strength over time
Figure 14. Total COD load and COD removal efficiency at various HRTs for the granular reactor.
Figure 15. Total COD load and COD removal efficiency over time for non-granular reactor
Figure 16. Total biogas production in (L/L/Day) at various HRTs for the granular reactor
Figure 17. Methane production in (L/L/Day) at various HRTs for the granular reactor
methane production for the granular reactor at various hydraulic retention times. These figures show the biogas and methane produced in liters per liter of reactor volume per day. The values for Figure 16 were calculated by averaging the daily biogas production during the week that the testing was being conducted at each HRT and dividing it by the reactor volume. These types of plots allow for biogas and methane production comparisons with reactors of other volumes. Both figures paralleled the changing system loads quite well. This indicates that the reactor was able to adequately handle the variations in the load caused by the changing leachate strength and lowering the HRT. Figure 17, which represents methane production, is roughly 67% of the total biogas production. Throughout this research the methane fraction of the total biogas was consistently between 0.65 and 0.70. This methane fraction is a typical value for anaerobic systems.

The total daily biogas and methane production for the non-granular biomass reactor are shown in Figures 18 and 19. The biogas production for this reactor was much more inconsistent than was the case for the granular reactor. The low loads that were applied to this system, coupled with the poor treatment efficiency, were the primary reasons that the biogas production was so much lower than for the granular reactor. As Figure 19 shows, the non-granular reactor was also able to maintain a methane fraction between 0.65 and 0.70 throughout its operation.

c) Mixed liquor suspended solids

The monitoring of the mixed liquor suspended solids (MLSS) within the reactors was one of the most important aspects of this study. One of the benefits of the ASBR is that it is able to hold solids better than many other treatment methods. Figure 20 shows how the mixed liquor suspended solids dramatically increased as the HRT was decreased for the granular reactor. This tremendous increase in MLSS was due to the build-up of inorganic particulates within the reactor. The precipitation of metals, primarily iron, was the cause of
Figure 18. Total daily biogas production in (L/L/Day) for the non-granular reactor
Figure 19. Methane production in (L/L/Day) for the non-granular reactor
Figure 20. Mixed liquor suspended solids at various HRTs for the granular reactor
this accumulation of inorganic material. The addition of sodium bicarbonate caused the pH of the leachate to rise to the point that the soluble metals formed inorganic metal carbonates and sulfides. These inorganic precipitates accumulated in the reactor causing the increase in MLSS. As Figure 20 indicates, the MLSS was able to increase to a concentration of over 100,000 mg/L. During this build-up of inorganics, the mixed liquor volatile suspended solids remained almost constant. This caused the percentage of volatiles within the reactor to decrease from 65% to 20%. This was an alarmingly low percentage of volatiles for this system. However, the extremely high solids content did not hinder reactor performance, but in a larger scale system, some type of solids control is definitely an operational aspect that would need to be considered.

Figure 21 shows how the MLSS within the non-granular reactor changed over time. This reactor experienced virtually the same phenomenon as the granular reactor only on a lower scale. The non-granular reactor was treating lesser amounts of leachate, so the opportunity for inorganic precipitates to accumulate within the reactor was not as high. It did, however, achieve similar reductions in the percentage of volatile solids within the reactor. The build-up of inorganics within this reactor did affect the performance of the system. The accumulating inorganics began to hinder reactor performance by forcing, or "crowding out" the active microorganisms. The inorganics were able to force the active mass out of the system because they settled better than the volatile solids.

d) Solids retention time

The solids retention time (SRT) within a reactor is the measure of how long the suspended solids remain within the reactor. As was discussed earlier, the commonly accepted minimum SRT for an anaerobic system operating at 35° C is 10 days. Throughout this study, the SRT of the granular reactor remained well above the minimum SRT. Figure 22 shows that at the start of the study, when the system was being operated at HRTs of 48 and 36
Figure 21. Mixed liquor suspended solids over time for the non-granular reactor
Figure 22. SRT at various HRTs for the granular reactor
hours, the granular reactor had an SRT in excess of 150 days. Due to the accumulation of inorganics within the reactor throughout the study, the SRT was calculated based on the volatile portion of the mixed liquor suspended solids. Figures 22 and 23 show the decrease in the SRT versus HRT and time, respectively, as the experiment progressed. This decrease in SRT occurred even though the MLVSS within the reactor remained almost constant through the all of the HRTs that were tested. Because the MLVSS remained constant, an increase in effluent volatile suspended solids was the cause for the decrease in SRT. This can be attributed to the large amount of inorganics within the reactor forcing the active mass out of the system. This "crowding out" did not adversely affect system performance. However, by looking closely at both Figures 22 and 23, it is obvious that if the current trend was to continue, the SRT would eventually become low enough to cause reactor failure.

The SRT of the non-granular reactor was affected in the same manner as for the granular reactor one. Figure 24 shows how the SRT for reactor two changed over time throughout its operation. The SRT increased until about week 18, after which it began to decrease rapidly. The decrease in SRT was caused by the "crowding out" of the active mass by the inorganics, as previously discussed. Although the SRT was still high enough for the system to operate properly, the extremely low amount of active mass within the reactor was unable to treat the leachate very efficiently.

The primary difference between the two systems was the starting MLVSS concentrations within the reactors. The tremendous concentration of volatile solids within the granular reactor (20,000 mg/L) allowed the system greater tolerance as the amount of inorganics increased inside the reactor. The non-granular reactor began with such a low MLVSS concentration (2,400 mg/L) that it was much more quickly and severely impacted by the accumulation of inert mass within the reactor.
Figure 23. SRT over time for the granular reactor
Figure 24. SRT over time for the non-granular reactor
e) pH, volatile acids, and alkalinity

The pH of both reactors was very consistent throughout the study. Due to the addition of the sodium bicarbonate buffer, there never was a time during the study when the pH dropped out of the preferred pH range of 6.8 to 7.2. The volatile acids present within the reactor effluent are an excellent indication of how well the methanogens are operating. If the volatile acids are low, then the methanogens are converting the majority of the acids into methane. Figure 25 shows how the volatile acids changed at various HRTs for the granular reactor. As expected, the volatile acids were low, indicating that the system was performing well. The line produced in this figure parallels the applied load line that was shown in Figure 14. The volatile acids present within the effluent of the non-granular reactor were much more inconsistent. This system's performance varied so much that pseudo-steady state was rarely achieved. Therefore, sufficient points to plot an adequate curve were not obtained. From the data that were taken it appeared that the volatile acids for this reactor generally paralleled the system load. The volatile acids for the non-granular reactor effluent ranged from to 274 mg/L to 480 mg/L as acetic acid.

The alkalinity within both reactors remained fairly consistent for the duration of the research. Figure 26 shows how the alkalinity varied as different HRTs were tested for the granular reactor. The alkalinity appeared to be independant of the applied HRT. However, the alkalinity did decrease as the load was increased, but this change was slight.

f) Automated image analysis

The automated image analysis (AIA) was performed on the biomass of both reactors three times during this project. The results of this testing proved to be inconsistent, but did indicate that a build-up of inorganic precipitates within the reactors was occurring. When the AIA testing was first conducted, it appeared that reactor one, which was seeded with granular sludge, was losing its granules as a result of the leachate substrate. This appeared to be
Figure 25. Volatile acids at various HRTs for the granular reactor
Figure 26. Alkalinity at various HRTs for the granular reactor
occurring because of a drop in the particle size distribution. However, further AIA tests proved that this was not the case. Rather, the reactors were so inundated with the small inorganics precipitates that it caused the particle size to decrease. This phenomenon became so severe that eventually the AIA was not able to analyze the samples because there was such a high concentration of small particles present in the biomass.

B. Phase Two Results

Phase two was conducted by operating the granular ASBR at a 24-hr HRT, collecting its effluent, and running it through the aerobic polishing unit that was previously described. This phase of the research lasted approximately 6 weeks.

This two stage treatment method produced excellent results. Figure 27 shows the load that was applied to the aerobic unit and the COD removal efficiency that this unit achieved. Figure 28 shows the total load applied to the two stage system and the overall COD removal efficiency that it was able to achieve. As this figure shows, the overall removal rates were very good.

Because the goal of aerobic treatment was to attain direct discharge limits, additional tests were conducted on the reactor effluent. Table 15 shows the results of these tests. This table indicates that the overall treatment of the leachate was excellent and that it is possible to achieve direct discharge requirements.
Figure 27. Total COD load and COD removal efficiency for the aerobic polishing unit
Figure 28. Total COD load and COD removal efficiency for the two stage system
Table 15. Concentrations\(^a\) of constituents tested on the final system effluent

<table>
<thead>
<tr>
<th>Testing parameter</th>
<th>Raw leachate</th>
<th>Final effluent</th>
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</thead>
<tbody>
<tr>
<td>TCOD</td>
<td>2,489</td>
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</tr>
<tr>
<td>SCOD</td>
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</tr>
<tr>
<td>BOD(_5)</td>
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</tr>
<tr>
<td>Ammonia-nitrogen</td>
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<tr>
<td>Total P</td>
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<td>0.5</td>
</tr>
<tr>
<td>TKN</td>
<td>----</td>
<td>24.4</td>
</tr>
<tr>
<td>Total Fe</td>
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<td>0.99</td>
</tr>
<tr>
<td>Total Mg</td>
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<td>1.01</td>
</tr>
<tr>
<td>Total Pb</td>
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<tr>
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</tr>
<tr>
<td>Total Mn</td>
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<tr>
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<tr>
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<tr>
<td>Volatile acids</td>
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</tr>
<tr>
<td>pH</td>
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<td>8.25 - 8.68</td>
</tr>
</tbody>
</table>

\(^a\) All concentrations except pH in mg/L
VI. CONCLUSIONS

The results of this research allowed the following conclusions to be drawn:

1. The preliminary results indicated that the anaerobic sequencing batch reactor could be developed into a feasible method for the treatment of landfill leachate.

2. Granular sludge would be recommended as the preferred reactor biomass based on its superior results.

3. The granular sludge was not adversely affected by the build-up of inorganic precipitates during this study.

4. Some type of solids control must be implemented to prevent reactor failure due to the problem of inorganic solids build-up.

5. Maintaining a constant HRT and allowing the system load to vary with the leachate strength did not adversely affect the granular reactor.

6. Aerobic polishing enables the achievement of direct discharge limits.
VII. RECOMMENDATIONS FOR FUTURE RESEARCH

The results and observations that were found throughout the course of this study yielded the following recommendations for future research in the area of leachate treatment with the ASBR system:

1. The loads that were applied to this treatment system were much lower than expected due to the severe flooding that occurred in the midwest the summer of testing. This study should be continued to see if the ASBR can successfully handle a stronger, more typical leachate.

2. A research project investigating various retention times for the aerobic polishing unit could be conducted.

3. Successful ASBR research has been conducted on swine wastes at lower temperatures. Because leachate is produced at remote locations, the investigation of leachate treatment at lower temperatures may be desirable.

4. Because this research seems so promising, research on a pilot scale ASBR could be investigated.

5. Although the granular reactor never reached failure, it was evident that the continued build-up of inorganics within the reactor would eventually lead to failure. A study could be implemented where the mixed liquor suspended solids are controlled within the system.
BIBLIOGRAPHY


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