Anaerobic sequencing batch reactor treatment of

swine wastes at 35° C and 25° C

by

Surya Raja Pidaparti

A Thesis Submitted to the

Graduate Faculty in Partial Fulfillment of the

Requirements for the Degree of

# MASTER OF SCIENCE

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

Signatures have been redacted for privacy

Signatures have been redacted for privacy

Ames, Iowa

1991

Copyright 🕑 Surya Raja Pidaparti, 1991. All rights reserved

# TABLE OF CONTENTS

Ι.	INTRODUCTION	1
<b>II</b> .	LITERATURE REVIEW	7
III.	EXPERIMENTAL SETUP	39
IV.	EXPERIMENTAL PROCEDURES	49
V.	RESULTS AND DISCUSSION	62
VI.	CONCLUSIONS	81
VII.	RECOMMENDATIONS FOR FURTHER RESEARCH	82
BIB	LIOGRAPHY	83
ACł	NOWLEDGEMENTS	88
APF	PENDIX A. GAS and pH DATA	89
APF	PENDIX B. CHEMICAL PARAMETERS DATA	97
APF	PENDIX C. SOLIDS AND SRT DATA	103
APF	PENDIX D. COD DATA	108
APF	PENDIX E. METHANE PRODUCTION DATA	113

.

# LIST OF TABLES

Table I.	Functions performed by methanogens	11
Table II.	Energy yielding reactions used by methanogens	12
Table III.	Stimulatory and inhibitory concentrations of common toxic materials	16
Table IV.	Manure production from growing and finishing swine	27
Table V.	Chemical composition of swine waste (dry weight basis)	28
Table VI.	Environmental characteristics of swine waste	29
Table VII.	Characteristics of raw swine manure and blended feed	50
Table VIII.	Reactor performance and monitoring parameters	51
Table IX.	GC operating conditions for gas analysis	56
Table X.	Total COD and volatile solids loadings at $35^{\circ}$ C and $25^{\circ}$ C	62
Table XI.	COD and gas production performance data at 35° C	64
Table XII.	COD and gas production performance data at 25° C	64
Table XIII.	Volatile solids performance data at 35° C	65
Table XIV	Volatile solids performance data at 25º C	65
Table XV.	SRT and system chemical parameters at 35° C	68
Table XVI.	SRT and system chemical parameters at 35° C	68
Table XVII.	Development of volatile acids in the incubated effluent sample	77

# LIST OF FIGURES

Figure 1.	Biochemical pathways of anaerobic digestion.	8
Figure 2.	Principle of an anaerobic sequencing batch reactor.	23
Figure 3.	Schematic of experimental setup.	40
Figure 4.	Schematic of reactor construction.	41
Figure 5.	Schematic of water lock displacement columns.	45
Figure 6.	Schematic of the wet tip gas meter.	48
Figure 7.	Reduction in total COD at temperatures of $25^{\circ}$ C and $35^{\circ}$ C.	66
Figure 8.	Volatile solids destruction at temperatures of $25^{\circ}$ C and $35^{\circ}$ C.	67
Figure 9.	Gas production at temperatures of 25° C and 35° C.	70
Figure 10.	Comparison of performance - ASBR versus conventional digesters.	71
Figure 11.	Solids retention times achieved at various COD loads.	74
Figure 12.	Log(MLVSS) - SRT plot for 35° C.	75
Figure 13.	Log(MLVSS) - SRT plot for 25° C.	76
Figure 14.	Recovery of gas production following shutdown.	78
Figure 15.	Flow scheme for swine waste treatment using the ASBR.	80

.

### I. INTRODUCTION

Advances in animal science and an ever growing demand for meat has led to the development and burgeoning growth of confinement livestock feeding facilities. Livestock, especially poultry and swine, in today's agriculture are bred and reared in indoor facilities. Such facilities house large numbers of animals under a single roof. Confinement facilities have become an accepted feature of animal husbandry, especially in the United States. Use of confinement facilities allows animal husbandry even in inclement winter months that could not have been possible otherwise.

#### A. Environmental Concerns

In lowa, about 15 million swine are raised in confinement facilities each year [1]. With these large scale operations, huge quantities of wastes comprised mainly of excreta, are generated and require disposal in an acceptable manner. It is estimated that a 100 lb animal generates approximately 7 lb wet weight of waste per day. This waste has a 5-day BOD of about 0.34 lb [2]. Therefore, the swine population in Iowa generates waste quantities that equal those generated by a human population of 26 million. A 1000 hog facility with an average animal weight of 100 lb would typically involve disposal of 7000 lb wet weight of solids or 340 lb of BOD<sub>5</sub> per day.

Waste from confinement feeding facilities, consisting of feces, urine and spilled feed, is extremely putrescent due to the gases generated by microbial degradation of the waste. Over sixty different gases and vapors, many toxic or irritating, are released from waste decomposition [3,4]. The gases include hydrogen sulfide, methane, carbon dioxide, ammonia and several mercaptans. Emission of these gases and vapors from decaying wastes poses significant

occupational and animal health hazards which include explosions of methane, irritation of the eyes, mucous membranes and respiratory tract from ammonia and hydrogen sulfide [3,5], acute toxic incidents (fatal and near fatal) and asphyxiation of the livestock from carbon dioxide [4,6].

Outdoor environmental concerns of odors and water pollution occur with land disposal of wastes from confinement facilities [7,8]. As mentioned earlier, odorous gases are generated from microbial action on the land spread wastes and cause public nuisances. Water pollution may result from runoff or surface contamination of groundwaters from the land applied wastes [2,9]. Stabilization of the waste prior to land application and proper incorporation into the soil could greatly reduce water and air pollution from such sources.

B. Swine Waste as a Low Intensity Energy Source

Swine waste is putrescent due to its highly biodegradable nature. It contains a rich supply of essential microelements iron, nickel, zinc and aluminium, in addition to high concentrations of starch, hemicellulose, proteins and lipids. The presence of these constituents makes swine waste a good candidate for biological stabilization. Biological stabilization can be applied using two processes - aerobic degradation or anaerobic degradation.

Aerobic degradation is a process carried out by bacteria, fungi and protozoa in the presence of oxygen [10]. The process usually results in the conversion of the organic substrates to new cells, carbon dioxide and heat. The microorganisms utilize the chemically bound energy in the substrates to synthesize new biomass. Approximately 59% of the energy is converted to cell mass, and the rest is used for cellular activity [10]. The process results in the production of large quantities of

waste biomass or sludge. The major disadvantages of the process are the generation of large amounts of sludge that require disposal, a high energy input needed to maintain aerobic conditions in the treatment unit, and frequent maintenance of the aeration equipment.

Anaerobic degradation is carried out by a select consortium of bacteria in the absence of oxygen. A major fraction (89%) of the energy in the substrate is converted to methane and released as an off-gas, and only 8% is used for synthesis of new cell mass [10]. Consequently the growth rates of anaerobic bacteria are low with regeneration times ranging from 4 to 10 days at 35° C [11]. Norman [10] cites a correct C:N:P ratio, high biodegradable organics content and a low redox potential as the chief factors for favoring anaerobic treatment to stabilize swine waste. Further, anaerobic stabilization leads to the recovery of a fuel gas, methane, rather than energy expenditure in aerobic stabilization.

Anaerobic treatment has been experimented with at both lab and pilot scales, and has been applied widely using various reactor vessel configurations. The various reactor configurations can be broadly classified into two groups:

- 1. Suspended growth systems
- 2. Attached growth systems

Suspended growth systems include continuously stirred reactors, plug flow systems and upflow sludge blanket reactors that employ bacteria freely floating in a liquid, to degrade substrates introduced into the liquid.

The bacteria exist as freely floating agglomerated masses or flocs. As there is a physical limit on the amount of solids that can be maintained in suspension, these systems are limited by the bacterial populations that can be maintained in the liquid. Due to the relatively small bacterial populations, these units are not very

resistant to shocks of organic load & temperature variations. Due to the suspended nature of the bacterial masses, a considerable mass of bacteria is lost when the treated effluent is wasted from the reactor.

Attached growth systems, on the other hand, employ bacteria partially immobilized on a stationary bed of inert media. Due to the large surface area of the bed media, these systems tend to accumulate much larger bacterial populations. Consequently, these systems can handle very high organic loads and are quite resistant to load and temperature shocks. However, due to the limited porosity of the bed material, these systems do not accept wastes with high suspended solids contents or coarse solids. Such wastes usually cause problems of clogging and fouling in attached growth systems.

Swine waste is characterized by a high solids content varying between 2% and 16% [10] depending on the quantity of water used for flushing of the facility. A typical solids content ranges from 4% to 6%. The high solids content and the coarse nature of the solids in the waste precludes the application of attached growth treatment systems. For the same reason, most of the applications of anaerobic processes to swine waste employ suspended growth systems.

Since suspended growth systems employ freely floating bacterial flocs, there is always a considerable loss of biomass along with the treated effluent. The rate of loss of bacterial flocs is defined as a retention time equal to the ratio of the bacterial solids present in the system to those lost each day. The rate of loss of bacteria from a suspended growth system defines its characteristics. While a rate of loss larger than the rate of regeneration implies a *washout* of the bacteria and imminent failure, a relatively small rate of baterial loss implies an increase in the average age of the cells. For anaerobic systems, rates of regeneration of the

bacteria are very low due to the meager assimilation of the substrate's energy. The time for cell replication, as it was stated earlier, ranges from 4 to 10 days at 35° C [11]. Therefore, treatment systems with less than ten days bacterial retention time eventually fail. Temperature has a profound effect on the regeneration rates. The rates change by a factor of two for every 10° C change in temperature [12].

Continuously stirred reactors, that employ continuous mixing of reactor contents, are the most commonly used reactor configurations for the digestion of municipal waste activated sludge. Due to the continuous mixing, these reactors have the same retention time for biomass as for the liquid. On the other hand, an Anaerobic Sequencing Batch Reactor based on the anaerobic activated sludge concept developed by Dague et al. [14,15], employs intermittent mixing coupled with phases of settling, effluent removal and waste inflow. Due to the periods of settling the biomass has a longer retention time than the liquid. It has been reported that intermittent mixing and effluent removal after a period of settling, prevents loss of microbial flocs and improves the efficiency of microbial degradation [14,15].

#### C. Objectives and Scope of Study

The purpose of this study was to establish that the performance and resilience of a completely mixed anaerobic reactor could be enhanced to a great degree by the introduction of intermittent mixing interspersed with periods of internal settling, effluent removal and influent introduction. This research was designed to demonstrate the enhanced ability of such a reactor to handle higher organic loads even at lowered temperatures. A study was conducted using three identical anaerobic reactors that were fed swine waste homogenized to about 2% solids content. The reactors were continuously mixed, by recirculation of the head

space gas through the reactor contents, during the mixing (react) phase. The performance of the reactors at several organic loads and two different temperatures was determined. A comparison of the performance of these reactors with that documented in literature at equivalent conditions was used to demonstrate the efficacy of the reactors. A comparison of the performance data at the two temperatures was used to establish the resilience of the reactors.

The objectives of this research can be summarized as:

1. To investigate the applicability of the anaerobic sequencing batch reactor (ASBR) to the stabilization of swine waste.

2. To investigate the effect of temperature on the treatment efficiency of the ASBR.

# II. LITERATURE REVIEW

#### A. Fundamentals Of Anaerobic Digestion

1. Biochemistry and Microbiology

Anaerobic degradation is the microbial conversion of organic carbon to methane and carbon dioxide in the absence of oxygen or oxygenated compounds such as sulfates and nitrates. The production of valuable methane as a byproduct renders anaerobic digestion an extremely attractive alternative for waste stabilization. Anaerobic degradation is carried out by a complex population of micro flora that release energy through the reduction of the organic carbon to methane [14]. The various bacteria are grouped into several functional groups or trophic levels depending on their catabolism of carbon.

Zeikus [15] has classified the bacteria into four trophic groups:

a. <u>Hydrolytic/ Fermentative bacteria</u> that catabolize polysaccharides, proteins and other macromolecular constituents of organic matter, to simpler molecules such as sugars, volatile fatty acids and amino acids.

b. <u>Hydrogen producing acetogenic bacteria</u> that catabolize certain fatty acids and neutral endproducts to hydrogen and acetate.

c. <u>Homoacetogenic bacteria</u> that catabolize unicarbon compounds such as formate and carbon dioxide to acetate and hydrolyze multicarbon compounds to acetate.

d. <u>Methanogenic bacteria</u> that catabolize acetate and unicarbon compounds to methane.

The interactions between the various groups of bacteria are represented in Figure 1. The stability of the entire anaerobic process is dependent on the degree



Figure 1. Biochemical pathways of anaerobic digestion

of coordination that is achieved between the various groups. Any environmental factor that influences the activity of one group can affect the entire bacterial population.

a. <u>Hydrolytic/Fermentative bacteria</u>: The hydrolytic/ fermentative group of bacteria are anaerobes that secrete extracellular enzymes that hydrolyze biopolymers and ferment the macromolecules to end products such as hydrogen, carbon dioxide, propionate, butyrate, other volatile acids and ethanol [16,17]. The bacteria in this group include both obligate anaerobes (e.g., <u>Clostridium</u>. <u>Bacteroides</u>, <u>Ruminococcus</u>, and <u>Butryvibrio species</u>) and facultative anaerobes (e.g., <u>Escherichia coli</u> and <u>Bacillus</u> species) [17].

Fermentative catabolism of most substrates proceeds by the Embden Meyerhoff Parnas pathway to pyruvic acid following hydrolysis. Pyruvate is further broken down to acetate, carbon dioxide and hydrogen, or to propionate and butyrate via lactate and succinate or to even ethanol. The pathway of pyruvate degradation is dependant on the partial pressure of hydrogen in the system. Hydrogen atoms are generated at various stages during the formation of pyruvate. Presence of excess hydrogen favors the formation of propionate, butyrate and ethanol. In an active and stable anaerobic process, hydrogen utilizing methanogens help maintain low hydrogen concentrations and promote formation of acetate, carbon dioxide and more hydrogen [14].

b. <u>Hydrogen producing Acetogenic bacteria</u>: These bacteria perform the following functions:

i. Oxidation of alcohols to the corresponding carboxylic acid, eg. ethanol to acetate and hydrogen.

ii. Beta oxidation of fatty acids with even number of carbon atoms to acetate, and of fatty acids with odd number of carbon atoms to acetate, propionate & hydrogen.

iii. Decarboxylation of propionate to acetate and carbon dioxide [18].

These organisms are also known as obligate proton reducers, since their major role is the oxidation of fatty acids and alcohols, and reduction of protons to molecular hydrogen [16]. Several of these acetogens have been documented. In each case, the acetogen exists in a syntrophic association with a hydrogen utilizing bacterium. The conversions of butyrate and propionate to acetate involve an increase in free energy and would not proceed spontaneously. However, the change in free energy for hydrogen utilization is extremely negative. Thus, when a hydrogen producing acetogen is coupled with a hydrogen consuming methanogen, the combined reaction becomes energetically favorable [14].

c. <u>Homoacetogenic bacteria</u>: These bacteria are mixotrophs that catabolize both hydrogen and carbon dioxide or multi-carbon compounds such as sugars to acetic acid. Some organisms of the <u>Clostridium</u> and <u>Acetobacterium</u> genera are homoacetogens. Though the role played by these bacteria is not exactly clear, their metabolism results in the maintenance of low partial pressure of hydrogen [15].

d. <u>Methanogenic Bacteria</u>: Methanogenic bacteria are obligate anaerobes that form methane as their metabolic end product [19]. These bacteria are the most important link in the consortium, as they are the only ones capable of:

i. using electrons in the form of hydrogen, andii. breaking down acetate anaerobically without exogenous electron acceptors.

Therefore, without methanogens, organic matter would not be degraded effectively, as organic acids with equal energy content would accumlate in the system.

Methanogens function as bioregulators of process stability and activity. They serve to regulate the flow of protons and electrons, and regulate nutrient levels. Table I. summarizes the functions of methanogens.

Function	Metabolic reaction	Process significance
1. Proton Cl Regulation	H <sub>3</sub> COO <sup>-</sup> + H <sup>+</sup> >CH <sub>4</sub> + CO <sub>2</sub>	1. Removes toxic protons
		2. Maintains suitable pH
2. Electron Regulation	4H <sub>2</sub> + CO <sub>2</sub> >CH <sub>4</sub> + 2H <sub>2</sub> O	1.Creates thermodynamically favorable conditions for catabolism of multi-carbon compounds by homo- acetogens.
		2. Prevents accumulation of H <sub>2</sub> and CO <sub>2</sub>
		3. Improves substrate utilization by hydrolytic bacteria.
3. Nutrient Regulation	Excretion of growth factors	1. Stimulates growth of hetero- trophs.

Table I. Functions performed by methanogens [20]

Of all the functions of methanogens, proton regulation is ecologically perhaps the most important function as:

i. the majority of the methane is derived from the acetate group which is significantly catabolized only by methanogens, and ii. high proton concentration can significantly inhibit  $H_2$  catabolism by both methanogens and acetogens.

iii. Decarboxylation of propionate to acetate and carbon dioxide [18].

The substrates that methanogens can use as both carbon and energy sources are limited to  $H_2/CO_2$ , formate, methanol, carbon monoxide, methylamine, and acetate [19]. Almost all methanogens can use hydrogen to reduce  $CO_2$  to methane. The methyl group in acetate accounts for 70% of the methane formed in nature. However, the change in free energy for the conversion of acetate to methane is a very small negative value [refer Table II]. The methanogens,

Reaction (	Go kJ/reaction)	Gº (kJ/ CH4)
4H <sub>2</sub> + CO <sub>2</sub> > CH <sub>4</sub> + 2H <sub>2</sub> O	-138.8	-138.8
4HCOOH>3CO <sub>2</sub> + CH <sub>4</sub> + 2H <sub>2</sub> O	-119.5	-119.5
4CH <sub>3</sub> OH> 3CH <sub>4</sub> + CO <sub>2</sub> + 2H <sub>2</sub> O	-310.5	-103.5
$4CH_3NH_3^+ + 2H_2O_{} + 4NH_4^+$	-225.7	- 75.2
4CO + 2H <sub>2</sub> O> CH <sub>4</sub> + 3CO <sub>2</sub>	-185.6	-185.6
CH <sub>3</sub> COOH> CH <sub>4</sub> + CO <sub>2</sub>	-27.6	-27.6

Table II. Energy yielding reactions used by methanogens [14]

G<sup>0</sup> is the change in Free Energy accompanying the reaction.

therefore grow slowly [19]. The replication time for each cell could be as high as 11 days at 35° C. Due to the small energy recovery in methane formation, methanogens are slow growing and are the most susceptible to environmental perturbations. They are perhaps the most critical part of the entire process. Failure

of an anaerobic system is often preceded by the failure of methanogens. Failure of methanogens leads to the rapid accumulation of propionate, butyrate and other intermediate electron products followed by a rapid decline in pH. All methanogenic bacterial activity is soon arrested.

**B.** Parameters influencing Anaerobic Digestion

# 1. Environmental Parameters

The various environmental parameters that affect anaerobic digestion are listed below [20]:

a. Temperature

b. pH, alkalinity & volatile acids

c. Toxic materials

The effects of these parameters on anaerobic digestion are described in detail in the following sections:

a. <u>Temperature</u>: The temperature at which the digestion process is operated has a profound influence on the rate as well as the degree of digestion achieved. In general, the rate of digestion increases with increasing temperature, approximately doubling for every 10° C rise [12]. However, it has been documented by many researchers that there exist certain distinct, optimum temperature ranges. Based on digestion studies at various temperatures from 10° C through 35° C, Rudolfs has pointed out that the rate of digestion, and not the total gas yield, is affected by temperature [21]. Heukelekian suggests the predomination of different flora at different temperatures [22]. Heukelekian and Heinmann reported on the growth of methanogens on synthetic cultures at various temperatures [23,24,25,26]. It was concluded that the optimum temperature for growth of methanogens was  $28^{\circ}$  C. The rate of growth and the ultimate yield were not significantly improved by a raise in the temperature to  $35^{\circ}$  C. However, a drop to  $20^{\circ}$  C, had significantly lowered the growth rate. The results strongly indicate the presence of an optimum from  $28^{\circ}$  to  $35^{\circ}$  C.

Heukelekian and Kaplovsky reported studies of digestion carried out at 50°, 40° and 20° C [27]. Sludge mixtures made with seed produced at 50° and 40° C were digested at 50°, 40° and 20° C. Temperature of some of the mixtures was lowered. The effect of lowering of temperatures was greater when the temperature was lowered from 50° to 20° C than from 50° to 40° C. Gas production was completely stopped at 20° C. It was concluded that the organisms responsible for digestion at 40° C were different from those at 35° C. The optimum for these bacteria was indicated as 50° C. The two temperature ranges were again confirmed by McCarty [28]. The lower one, from 28° to 35° C was termed as mesophillic range and the higher one from 50° to 70° C as the thermophillic range.

b. <u>pH. volatile acids and alkalinity</u>: A proper pH is one of the most important environmental requirements for successful anaerobic treatment. Each of the bacterial groups that constitute the digester population have different optimal ranges of pH. lanotti and Fischer report on the pH range for initiation of growth of hydrolytic/ fermentative bacteria [29]. A pH range of 6.2 to 9.1 permitted growth of two species, Peptostreptococcus I & II, while a pH above 8.2 inhibited another species. Seagren [20] has pointed out that all methanogens have optimum pH ranges between 6 to 8.

In mixed populations comprising bacteria from all three groups, the optimum pH has to accommodate all of the involved species. Work done by Clark

and Speece [30] takes a comprehensive look at the effects of adverse pH levels of 3.8 to 9.4. It was found that steady state methane production occurs at pH levels as low as 4 ; however the rates are lower than for the same reactor operating at optimum pH. No inhibition of methane production was observed between pH 6 & 8. Temporary pH shocks did not have long lasting effects. McCarty recommended a pH range of 6.6 to 7.6, with an optimum of 7.0 to 7.2 [28]. The USEPA' s <u>Operations Manual Anaerobic Sludge Digestion</u> reports that a healthy digester has a pH in the range of 6.8 to 7.2 and plenty of buffering alkalinity.

The maintenance of a high level of alkalinity is important for process control, since it represents the ability of the system to neutralize acids that are formed during anaerobic digestion or are present in the influent. The bicarbonate alkalinity is the predominant form, since it has a  $pK_a$  near 7.0 and is present in significant concentrations.

The total volatile acids present in the system is also an important system environmental parameter. High levels of volatile acids can be tolerated if the acidity is neutralized with a cation of low toxicity [28]. The USEPA <u>Operations</u> <u>Manual Anaerobic Sludge Digestion</u> recommends use of the ratio of total volatile acids (TVA, in mg/L) to total alkalinity (TAlk, in mg/L), TVA / TAlk, as the major process control parameter. In an upset digester, the TVA will increase followed by a decline in TAlk. The ratio would therefore emphasize these changes, indicating a process upset.

c. <u>Toxic materials</u>: There are several materials that are inhibitory or toxic to anaerobic processes. The toxic materials include, alkali and alkaline earth salts, ammonia, sulfides, heavy metals and several organics. A comprehensive summary on toxic materials and their control has been presented by McCarty [31].

Most toxic materials are stimulatory at low concentrations but turn inhibitory as the concentrations increase. Table III lists the stimulatory and inhibitory concentrations of various materials. Alkali and alkaline earth metals are usually added to digesters to improve pH control.

Material	Stimulatory	Moderately Inhibitory	Strongly Inhibitory
Sodium	100-200	3500-5500	8000
Potassium	200-400	2500-4500	12000
Calcium	100-200	2500-4500	8000
Magnesium	75-150	1000-1500	3000
NH <sub>3</sub> -N	50-200	1500-3000	>3000

Table III. Stimulatory and inhibitory concentrations<sup>a</sup> of<br/>common toxic materials [31]

<sup>a</sup> All concentrations are in mg/L

The concentrations classified as moderately inhibitory are those which can be tolerated but require some acclimation of the micro-flora. When introduced suddenly, these concentrations can be expected to retard the process significantly for a brief period of time. On the other hand, the concentrations listed as strongly inhibitory are those which will retard the process to such an extent that the efficiency will be low.

Ammonia is usually formed in anaerobic processes as a result of reduction of organic nitrogen in wastes rich in protein or urea. It may be present either in the form of ammonium ion  $(NH_4^+)$  or as dissolved ammonia  $(NH_3)$  gas. The two forms

are in equilibrium with each other, the relative concentrations being dependant on the pH or hydrogen ion concentration as indicated by the following equation:

 $NH_4^+ < NH_3 + H^+$ 

When hydrogen ion concentration is sufficiently high (pH of 7.2 or lower), ammonium ion predominates. At higher pH levels, the equilibrium shifts to the right with ammonia being predominant. Ammonia is inhibitory at much lower concentrations.

Sulfides in anaerobic digesters may arise from the organic substrate and the reduction of sulfates in the influent. Sulfides exist as soluble sulfide ions, insoluble precipitates of metals and as dissolved hydrogen sulfide gas. The actual distribution is pH dependant . Concentrations of soluble sulfide from 50 to 100 mg/L are tolerated with little or no acclimation. These concentrations are actually desirable as they help remove heavy metals from the digester. Concentrations up to 200 mg/L are tolerated after some acclimation. Concentrations above 200 mg/L are quite toxic [31].

There are many toxic organic materials that may inhibit the digestion process. These range from organic solvents to common materials such as alcohols and long chain fatty acids. Examples of such materials are methanol (toxic above 1000 - 2000 mg/L), sodium oleate (toxic above 500 mg/L). Most organics can be treated by using a continuous feed scheme and lowering instantaneous concentrations. Sodium oleate toxicity is reduced by precipitation of oleate with calcium chloride. Some detergents are also toxic or inhibitory.

2. <u>Operational Parameters</u>

The operational parameters that influence anaerobic digestion in suspended growth systems are listed below:

a. Solids Retention Time

b. Organic Loading Rate

c. Hydraulic Retention Time

d. Mixing

a. <u>Solids Retention Time</u>: Solids retention time (SRT) has been used as a key parameter in the design and operation of anaerobic treatment processes for a long time. Under steady state conditions, the SRT is defined as follows [12]:

# SRT = <u>Total Biomass in the system</u> Biomass wastage per day

For succesful operation of any system, the SRT should be longer than the regeneration time of the slowest growing organisms in the system or the system eventually fails from washout of the bacteria. If the slowest growing organisms have a unique role that no other species can perform, the washout causes a loss of the function and a disruption or failure of the process. This leads to the accumulation of intermediates, many of which are inhibitory. The entire process could eventually fail from the mounting inhibition.

The rate of regeneration depends on various factors. The factor that influences the regeneration rate most is the temperature at which the organisms are maintained. The regeneration rates double for every 10° C rise in temperature following approximately Arrhenius Law [13]. Since the regeneration rate is temperature dependent, the regeneration rate and the minimum SRT required are also temperature dependent. At 35° C, the minimum SRT recommended is 10 days. Below an SRT of 10 days, it has been reported by McCarty [32] and, Speece and McCarty [33] that organic removal efficiencies drop off and intermediates such as propionate accumulate rapidly between a SRT of 4 to 8 days at 35° C. Dague et

al. [12] illustrated the effect of lowering the SRT below 10 days at 35° C. The total gas production, methane production and the percent removals of VS and BOD declined rapidly below this value. On the other hand, as the SRT is increased, the percent removals increase. The influent organic material is stabilized to a greater extent. This has been clearly demonstrated in plots by Dague et al [13]. It has been pointed out that the detention time required for a stable digestion is dependant on the temperature of digestion. A minimum detention time of 20 days at 25° C, 10 days at 35° C, and 5 days at 45° C is recommended. It is also pointed out that the regeneration rate of the slowest growing methane formers is about 10 days at 35° C. Below a solids retention time of 10 days, gas production declines rapidly due to the washout of key methane formers. Complete failure occurs at a detention time of about 3 to 4 days. It is stated that the effects of temperature changes on removal efficiency can be counteracted by varying the SRT. The reduction in performance due to lowered temperature can be eliminated by increasing the SRT. This is a key factor that calls into question the earlier reports, on temperature optima, cited on page 14 of this report. Early research on anaerobic degradation really did not consider population dynamics of the methanogens.

b. <u>Organic Loading Rate</u>: The organic loading rate represents the quantum of the organics that must be handled by the system. Measured in units of mass of volatile solids influent to the system per unit system volume per time, this parameter is an index of the stress imposed on the microbial population. The effects of organic loading on system parameters such as total gas and methane production, volatile solids destruction, COD stabilization and alkalinity were investigated by Pfeffer et al [34]. The alkalinity increases with increasing load, but the alkalinity developed at a given load is directly dependent on the temperature.

The higher the temperature, the greater the alkalinity. This was attributed to the temperature effect on the solubility of carbon dioxide and, therefore, a greater loss of carbon dioxide in the liquid effluent. The increase in alkalinity with increase in load is attributed to the increased formation of ammonium bicarbonate. Ammonium bicarbonate forms a substantial portion of the alkalinity.

The methane production per unit unit of volatile solids destroyed is nearly constant with increasing load as the destruction of volatile solids represents a loss of energy from the system and appears as methane production. Although methane production is constant, the total gas production increases with increasing load. With increasing load, the total production of all products of degradation increases per unit liquid volume of reactor. The total gas production increases, as the solubility of carbon dioxide is a function of its partial pressure and not the amount of the total gas production. However, the percentage of the influent organics that are destroyed declines with increasing load.

c. <u>Hydraulic Retention Time</u>: Hydraulic retention time or HRT is a measure of the rate of liquid flow into and out of a reactor. Under steady state conditions, HRT is defined as follows:

HRT = Total volume of liquid in the system Volume of liquid changed per day

In a completely mixed system that employs continuous mixing, all the contents of the system have the same residence or retention time. In such a system, the detention time is governed by the replication time of the slowest growing organism of the microbial population. Below this value, the system fails from washout of the slowest growing organism. On the other hand, in systems such as the ASBR and the upflow sludge blanket reactors, the SRT is delinked from HRT

through internal settling and biomass retention. HRT can be varied independant of SRT. It represents the magnitude of daily dilution taking place in the reactor. The greater the HRT, the greater the dilution. While dilution of the reactor contents reduces the concentrations of inhibitors and improves process efficiency, a larger flow rate also represents a faster rate of substrate removal from the reactor. The first effect is more pronounced at low HRTs, but as the HRT is increased the second effect tends to predominate and process efficiency may decline.

d. <u>Mixing</u>: Mixing is generally considered essential to efficient anaerobic waste treatment [13]. Mixing facilitates good contact between the substrate and the microorganisms and results in uniform conditions of substrate, temperature, hydrogen ion concentration, and the concentration of inhibitors. Taiganides has reported system failure resulting from localized accumulation of fatty acids and ammonia in pockets of high fiber concentrations [2].

However, should be applied only to achieve periodic turnover of the reeactor contents. Dague et al. [13] have reported on the effects of mixing in anaerobic systems. Intermittent mixing resulted in an increase in the gas production and in increased COD and solid removals compared to values obtained in continuous mixing [13]. It was suggested that mixing affects bioflocculation. Intense, continuous mixing was found to cause dispersed microbial growth, while intermittent mixing resulted in a readily settleable floc. The improved settleability of the biomass results in improved biological solids retention and increased methane production.

### C. Fundamentals of Anaerobic Sequencing Batch Reactors

The anaerobic sequencing batch reactor is based on the aerobic activated sludge concept developed by Dague et al [12,13]. The principle of operation is illustrated in Figure 2. The operation consists of a set of four sequencing phases. In phase I (the fill phase), the waste stream is introduced into the reactor and mixed with the biomass. Phase II (the react phase), is a phase where intimate contact between the microorganisms and the waste is maintained by continuous or intermittent mixing. The microbes utilize the substrates in the waste and produce methane. In phase III (the settle phase), the mixing is stopped and the solids are allowed to settle. The supernatant is removed from the reactor and another cycle is started. The anaerobic activated sludge concept was first applied as an anaerobic contact process to the treatment of meat packing wastes [35,36,37,38]. The contact process was capable of achieving high BOD removals at short liquid retention times. The application of the contact process to sewage, was limited due to the gasification in the settled sludge and separation of solids.

On the other hand, Dague et al. [12] report the development of a readily settleable sludge that did not gasify during sedimentation. It was concluded by Dague et al. [12] that the settleability of the sludge is dependant on the food to microorganism ratio maintained in the system. At low organic loads, the system operates in the same manner as the extended aeration activated sludge process. Under such conditions, the metabolism of the substrate is complete and most of the microbes are in the endogenous phase and good settling takes place. The presence of anaerobic protozoa under such conditions has been reported [13]. Protozoa are believed to reduce the turbidity by removal of dispersed growth, in a manner similar to the aerobic activated sludge process.



Figure 2. Principle of an anaerobic sequencing batch reactor

•

It is also reported [12] that the anaerobic activated sludge system is capable of operating at lower temperatures without loss in activity. The efficiencies of removal and gasification at 25° C were equal to those achieved at 35° C. These results indicated the ability of the system to counteract the effect of lowered temperature.

At lower temperatures, the solids content of the system was found to increase. The increase in the solids content was attributed to the increase in the microbial population brought about by lowered endogenous decay rates. Therefore, with good solids retention, it is possible to build a larger microbial population. Operation of the system with low F/M ratios allowed good solids retention and long SRTs . The increased microbial populations are reported to compensate for the decline in activity & metabolic rates due to the lowered temperature.

The performance of the system is affected by the mixing applied in the react phase and the solids retention time (SRT) achieved. Excessive turbulence and SRTs lower than the regeneration time of the microorganisms result in the dispersion of organisms and poor solids separation.

The performance of the system is reported to be enhanced by the chemical precipitation and flocculation of the dispersed growth in the effluent and its subsequent return to the system after settling [13].

## D. Review of Literature on Swine Waste Chracteristics

Hog wastes generated from confinement facilities represent a serious threat of odor and water pollution in the absence of stabilization and efficient disposal. In order to plan and design a method of manure disposal, it is essential that the

quantitative and qualitative characteristics of the manure are known and are accounted for in the design. The quantitative aspects would include the total mass, volume and water content of the waste requiring disposal. Some of the important qualitative parameters influencing the design are the organic content, the pH and the total nitrogen content. Both qualitative and quantitative aspects are influenced to a large extent by the animal characteristics, the feed rations and the environmental conditions in the confinement facility.

#### 1. Quantitative aspects of manure disposal

The quantity of waste to be disposed of is directly dependent on the amount of fecal matter and urine that is excreted by the animals. The quantity of fecal matter and urine excreted by a hog is, in turn, dependent on its metabolism of the feed and the environmental factors. The metabolism of the feed varies with the age of the animal, commonly measured in terms of its weight. The weight gained per day is an approximate measure of the metabolic rate of the animal [39]. Hazen and Mangold [40], using the data on the environmental conditions in the confinement feeding facilities and feed rations, estimated the variations in the daily manure production to be expected with the growth of the animals. They calculated the average daily manure production per hog to be one tenth the live weight of the animal. The amount of manure increases as the weight of the animal increases and the urine content is always more than 50% of the total weight. The manure production, is therefore directly related to the feed intake, water intake and age (or weight) of the hog.

The quantities of manure are influenced not only by the age of the hog and the feed, but by the environment as well. The environmental factors include the amount and type of bedding used, the humidity and temperature of the ambient air

in the building. In a facility, where straw is extensively used for bedding, the quantity of waste is significantly increased by inclusion of straw from the bedding. On the other hand, if no bedding is provided, the manure would not contain straw and the quantities would be reduced. The ambient temperature and humidity have significant effect on the manure quantities. Both the factors control the amount of water lost from the hog through exhaled air as well as from the manure through evaporation. Taiganides [39] reports that the manure quantities from a confinement facility are greater in winter than in summer. The decline in the manure production was accounted for by the increased amounts of moisture removed by the ventilation system in warmer weather.

Taiganides has presented a theoretical basis for estimation of manure quantities. The manure quantity is related to the feed and water intake, the heat and moisture loss to the environment from the hog and the manure, and the weight gain. A mass balance is presented to relate all of these quantities. Taiganides concludes that the water intake and moisture loss are perhaps the greatest sources of error in the estimation and need to be measured accurately. An average value of 4.7 (5.0 for design) lbs wet weight per 100 lb live weight is suggested by Taiganides for the purpose of estimation. In another paper by Taiganides and Hazen [41], the value suggested is 7 lb for every 100 lb live weight. Dague [42] has cited values suggested by Taiganides and Hazen as well as those cited by Irgens and Day [43]. The values cited from Irgens and Day are reproduced in Table IV. Based on the values suggested by Taiganides and Hazen [41], quantities of manure to be to be considered for disposal are listed by Dague [42]. The manure to be disposed of for every thousand head of swine, with a final weight of 200 lbs, is 14,000 lb or 7 tons wet weight per day.

Feces Ib	Urine Ib	Total Manure Ib	Total Volume gallons
0.7	1.5	2.2	0.36
2.7	2.9	5.6	0.67
5.4	6.1	11.5	1.38
6.5	8.1	14.6	1.75
8.5	9.1	17.6	2.11
	Feces lb 0.7 2.7 5.4 6.5 8.5	Feces lbUrine lb0.71.52.72.95.46.16.58.18.59.1	Feces lbUrine lbTotal Manure lb0.71.52.22.72.95.65.46.111.56.58.114.68.59.117.6

Table IV. Manure Production from Growing and Finishing Swine [39]

#### 2. Qualitative aspects of swine waste disposal

Pig waste is highly polluting due to its large content of easily degradable organic matter. The organic matter undergoes incomplete microbial decay, with the evolution of malodorous intermediates, in the absence of proper stabilization and disposal. The feces in the manure are composed of the undigested portions of the feed intake, bacteria carried out from the digestive tract, digestive enzymes and water. Since the breakdown of the feed in the alimentary canal is quite small (low digestive coefficients of 0.57 for fiber, etc. [44]), feces contain a large number of ingredients in original their form as well as the products of digestion.

The general constituents of swine waste are cellulose, hemicelluose, lignin, proteins and lipids. Starch is not usually found, as it is easily assimilated by the animal. A typical composition of the waste is listed in Table V. Most protein, vitamin and mineral additives to the feed are usually absorbed in the alimentary canal, but are excreted in the urine.

From a point of view of disposal or stabilization of the waste as well as that of pollution, some of the important parameters are the biochemical oxygen demand (BOD), the chemical oxygen demand (COD), and the ammonia content.

Component	Concentration
Cellulose, % TS Hemicellulose, % TS	11.4
Lignin, % TS	69.0
Protein, % TS	18.9
Lipids, % TS	13.7
Total Nitrogen, % TS	5.3
Ammonia Nitrogen, % TS	2.2
Ash, % TS	14.0
Calcium, % TS	3.2
Phosphorus, % TS	1.7
Potassium, % IS	0.9
Magnesium, % 15	0.8
Sulfur, % 15	0.3
Zing norm	249.0
	10/0 0
	544.0
Managanese nom	342.0
Cobalt, ppm	6.1
Molvbdenum, ppm	0.3
Cadmium, ppm	1.0
Lead, ppm	12.1
Gross energy content, MJ/Kg	18.1

Table V. Chemical Composition of Swine Waste (dry weight basis)[10]

Norman [10] has summarized qualitative analyses reported by various researchers. The salient features of the summary are reproduced in Table VI. The parameters vary over a wide range. The variation can be related to factors such as environmental conditions in the facility, feed rations and age of the animals. This is especially true of most confinement facilities wherein animals of different ages are maintained in the same facility and are fed different feeds. Often, different ambient temperatures are maintained for each age group in order to achieve optimal growth of the animals at each stage. The variation in waste characteristics with age

Parameter	Range
Chemical Oxygen Demand, gm/L	17.1-125
BOD <sub>5,</sub> gm/L	5.4-28.4
Total Solids, gm/L	16.5 -115
Total Volatile Solids, gm/L	14.4 - 93
Total Suspended Solids, gm/L	13.7 - 56
Volatile Suspended Solids, gm/L	11.4 - 13
Total Kjeldahl Nitrogen, gm/L	1.27 -6.9
Ammonia Nitrogen, gm/L	0.59 - 4.15
Alkalinity, gm/L	8.49 -10.4
Acetic acid, gm/L	210 - 2418
Propionic Acid, gm/L	70 - 675
рН	6.2 - 7.1
COD	2.49 - 4.28
BOD <sub>5</sub>	

Table VI. Environental Characteristics of Swine Waste [10]

has been illustrated well by the research done by Donham et al [45]. Extensive sampling of manure pits below the confinement buildings was conducted. A comparison of the waste characteristics indicated that the manure concentrations increased with swine age from farrowing to nursery to finish. However, the manure from the farrowing had a significantly higher sulfide content. The wastes are also affected to a large extent by the manure removal practices employed at the facility. A facility where manual scraping and hosing with water is practiced, the wastes are generally more concentrated than one where hydraulic flushing is used.

One obvious characteristic of swine waste is the offensive odor. Some of the odorous components of swine waste are:

a. hydrogen sulfide,

b. ammonia,

c. aromatic amines such as indole and skatole,

d. volatile fatty acids such as butyric acid, propionic acid and iso-valeric acid,

e. aromatic compounds such as phenols, cresols, benzoic acid, etc.

### 3. <u>Bacteriology of Swine Manure</u>

The bacteriology of swine manure is largely dependant on the feed given to the animal and the age of the manure at the time of sampling. A large population of anaerobic bacteria, as many as  $10^8$  to  $10^9$  per mL. of manure diluted to 4% solids, is present in the fecal matter [46]. This figure includes both facultative and obligate anaerobes. Three main morphological forms have been reported [46,47,48]:

a. <u>Facultatively anaerobic *Streptococci*</u> These comprise 43 - 74% of the entire bacterial population. These bacteria are gram positive, spheres that have been isolated as pairs or chains. The exact role played by these bacteria is not clear. No cellulolytic, proteolytic or amylolytic activity has been associated with these flora. It is believed that they may be responsible for the maintenance of a low hydrogen concentration and therefore favoring methane formation.

b. <u>Obligately anaerobic *Clostridia*</u> These are gram positive, motile rods with subterminal spores. These bacteria fermented mono-, di-, and polysaccharides to acetic and butyric acids.

c. <u>Obligately anaerobic</u> Bacteriodes. These are Gram negative pleomorphic rods. These bacteria ferment a variety of mono- and di-saccharides and most can degrade starch. None of the bacteria in this group are capable of cellulose or protein hydrolysis.

## E. Application of Anaerobic Digestion To Swine Waste

Anaerobic digestion has been applied to swine waste at both laboratory and pilot scales. As explained in an earlier section, the applications can be classified as suspended growth systems and attached growth systems. A summary of the research reported and the general results is presented in this section. A detailed summary of investigations on the effects of parameters such as organic loading rate, temperature, etc., on anaerobic treatment in suspended growth systems, is also presented.

### 1. Attached Growth Systems

Attached growth systems are systems, wherein the organisms responsible for the degradation, exist as layers of biological growth attached to or suspended in a bed of biologically inert support media. Stationary beds of media commonly termed as filters, are the most widely used attached growth systems. The bed of inert media serves to hold the organisms in the system. The bed may be composed of natural media such as fragmented rock or plastic. Use of synthetic polymer media allows precise control over bed porosity and surface area to meet design requirements.

The fine porous nature of the bed limits the type of waste streams that can be handled by an attached growth system. Waste streams with little or no particulate matter are handled best by attached growth systems. Waste streams with coarse particulate matter present problems of clogging and fouling of the fine porous beds. The main advantage of attached growth systems is the ability to accumulate large bacterial populations. The presence of a large population enables the system to handle high organic loads as well as withstand shocks of load, shutdown and sudden temperature changes. Some of the biomass is present as freely floating

mass in the interstices of the bed. With continual operation, the floating biomass tends to accumulate in the lower portions of the system. Therefore, there exists a gradient of decreasing biomass concentration from the lower portion to the upper portion of the bed. Most anaerobic filters utilize this distribution by the introduction of the waste stream at the bottom and removal at the top.

Application of anaerobic filters to the stabilization of swine waste has been limited. Swine waste contains significant concentrations of coarse solids and poses a threat of frequent clogging in filters with static beds. There have been, however, a few attempts to treat screened and settled swine waste in anaerobic filters [49, 50,51,52,53].

Brumm & Nye [49] have reported the use of swine waste, screened through a 1.19 mm. mesh, as a feed for anaerobic filters. The filters were operated at 24° C and, HRTs of 1, 2, 3 and 6 days. The organic loads achieved were 0.65, 1.12, 1.91 and 3.99 g COD/L/d. They report volatile solids destructions ranging from 46% to 58 %. The percent organic removals determined for swine waste treatment in anaerobic filters are cited to be lower than those reported for substrates such as brewery wastes and synthetic carbohydrate - protein wastes. The removals achieved are also reported to decline with increasing load and faster rates of flow through the filters.

Investigations on anaerobic filter treatment of swine waste have also been reported by Ng & Chin [50]. High volatile solids removals of 90% to 99.5%, and high COD removals of 84% to 97% have been reported at various organic loads and HRTs for a filter treating swine waste. The waste was screened through a 2 mm. sieve prior to feeding. The filter had a bed composed of lengths of PVC pipes and was fed intermittently with an upward flow. The effluent had BODs lower than
100 mg/L for HRTs greater than 2.8 days. The methane content of the gas recovered from the filter was dependent on the liquid flow rate in the filter. The higher the flow rate, the lower was the methane content of the gas recovered.

Use of expanded bed filters has been reported by Ng and Chin in another paper [51]. Two filters, one with a granular activated carbon (GAC) bed and the other with a sand bed, were used. Swine waste used as a feed was screened through a 2 mm. sieve prior to feeding. The filters were fed on an intermittent basis and were operated at 30° C at detention times varying from 2 to 6 days. Stable COD removals were obtained at HRTs ranging from 18 hours to 6 days. The COD removals had a larger range of variation in the sand filter than in the GAC filter. It was suggested that the GAC filter was capable of a better substrate retention by adsorption and hence good removals irrespective of the load. The GAC filter was, however, beset with problems of attrition with large concentrations of fines being washed out along with the effluent. Reduction of HRT worsened the problem of attrition, but the gas quality and production were unaffected. It is suggested by the authors that the expansion of the bed at the time of feeding allowed for redistribution of solids in the filters and prevented clogging [51].

Colleran et al. report the digestion of pig waste in an anaerobic filter following gravity settlement and liquefaction [52]. Pig waste slurry with a COD of 200 g/L and a solids content of 13% was allowed to settle and liquefy for a period of 12 to 15 days. The resulting supernatant had a COD between 10 g/L and 60 g/L, and a solids content of less than 1%. The supernatant was treated in four anaerobic filters, each with a different media, operated at 30° C. The COD removals achieved were identical for the different filters and averaged 69% at a COD load of 4.8 g/L/d. In a pilot scale filter operated at 28° C and a load of 19.6

g/L/d, the COD removals averaged 88%. It is reported that the filters were not affected by sudden changes in feed strength and handled shutdown periods without loss of efficiency.

Wilkie and Colleran report pilot scale digestion of pig slurry supernatant using an upflow anaerobic filter in another paper [53]. Gravity settled pig slurry was digested in an upflow anaerobic filter containing polypropylene support media at various organic loads and various HRTs. The solids content of the settled slurry varied from 0.7% to 1.79%. At an average COD load of 2.2 g/L/d, a temperature of 25° C and a HRT of 6 days, the filter yielded an average COD removal efficiency of 66%. The COD removal efficiency was unchanged even when the load was increased to 4.3 g/L/d. The COD removal efficiency is reported to drop to 52% on reducing the HRT to 3 days and increasing the load to 8.4 g/L/d. In a third set of experiments the temperature was raised to 35° C, and a removal efficiency of 60% was achieved at a load of 9.9 g/L/d.

#### 2. <u>Suspended Growth Systems</u>

Systems that comprise of masses of microorganisms freely floating in a liquid can be classified as suspended growth systems. These systems are not restricted by the particulate nature of a waste stream. However, these systems are restricted by the concentration of suspended solids that can be held in a reactor. These systems have been widely experimented with and applied to the treatment of swine waste. Several parameters affect the perfomance of these systems. The parameters include organic loading rates, detention times, and temperature. A summary of the parameters and their effects on anaerobic digestion of swine waste in suspended growth systems is presented in the following sections.

a. <u>Influence of Organic Loading Rate</u>: Organic loading rate represents the stress imposed on the microbial population in the system. The greater the stress, the lower is the removal efficiency. BOD removals from 80 to 90%, and COD removals from 50 to 70% at organic loads from 0.08 to 0.2 lb Volatile Solids/ cu. ft./ day have been reported by Hobson and Shaw [47].

Anaerobic digestion at various organic loads has been reported by Taiganides [39]. Digestion was carried out in completely mixed digesters at loads from 0.05 to 0.243 lb VS/ cu. ft./ day. Stable digestion of manure, could not be sustained above a load of 0.1 lb VS/ cu. ft./ day. All the digester failures were characterized by the presence of high alkalinity and ammonia levels, a rapid decline in gas production and a pH above 7.0. The inability of the digesters to handle loads higher than 0.1 lb VS/ cu. ft./ day, was attributed to the build up of high levels of ammonia and copper in the digester fluid. Copper is a feed additive given to the animals and, the feces contain as much as 1000 ppm of copper on a dry weight basis. High levels of copper and ammonia are cited to be severely inhibitory or toxic to anaerobic digestion. Accumulation of fibrous materials is also believed to cause localized build up of volatile acids leading to acidic conditions.

Stable digestion has been reported at significantly higher loading rates by Fischer [54]. The completely mixed digesters were fed swine manure at a 15 day HRT and varying solids concentrations. At a constant HRT, the manure concentrations represent loading rates. Manure concentrations, corresponding to loading rates as high as 0.4 lb VS/ cu. ft./ day, have been reported to be feasible. Digester failure has been reported only at a solids concentration of 107 g/L (equivalent load= 0.45 lb VS/ cu. ft./ day) and was characterized by a sharp decline in gas production. Significant solids removal efficiencies from 60 to 35% have

been reported for stable digestion. A similar rapid decline in gas production and conversion efficiency at 6% total solids (TS) in the input (equivalent load= 6 g TS/L/d) at 35° C, has been reported by Summers and Bousfield [55]. Stable digestion of swine manure at a load of 5.4 g VS/ L/d (= 0.34 lb/ cu. ft./ d) at 30° C, has been reported by Van Velsen [56]. It is reported that the digestion was stable, in spite of ammonia concentrations reaching 1600 to 4000 mg/L at a pH of 7.9.

b. Influence of Temperature: The effect of temperature on the anaerobic digestion of swine waste in continuously and completely mixed reactors or CSTRs have been reported in detail. It has been reported by Van Velsen et al. [57] that no gas production occurred in seeded anaerobic digesters at or below 13° C. Gas production ceased as the temperature was reduced to 13° C, while maintaining the other parameters constant. An increase in the volatile acids concentration was also observed. A closer examination of the data revealed that while the methane formation had ceased, the hydrolysis and acid formation continued resulting in higher volatile acids concentrations. The critical temperature at which stable anaerobic digestion can be initiated and sustained was reported by Zeeman et al. [58] to be between 5° C and 10° C. Initiation of digestion at the critical temperature was characterized by a lag period of 30 - 40 days. The lag period was dependant on the type and amount of seed used.

Psycrophillic or low temperature digestion, in the sediments of an anaerobic lagoon treating swine waste, was studied by Cullimore et al [59]. Initiation of anaerobic digestion was found to occur between 0° C and 8° C. However the temperature at which gas production started has been reported to progressively decrease with acclimation. It has been suggested that acclimation of the digester allowed development of bacteria capable of digestion at low temperatures.

Research on digestion of swine waste at temperatures in the range of  $45^{\circ}$  C to  $55^{\circ}$  C, or the thermophillic range, has been limited. Van Velsen et al. [57] have reported anaerobic digestion of swine waste at  $55^{\circ}$  C. The gas production, at similar conditions of organic load and detention time, is reported to be lower in the thermophillic range. It is suggested that high levels of molecular ammonia, formed by increased dissociation of ammonium ion, increasingly inhibit the digestion causing a lower gas production.

A mechanism of ammonia inhibition at thermophillic temperatures has been suggested by Wiegant and Zeeman [60]. High levels of molecular ammonia leads to the inhibition of hydrogen consumption. This results in inhibition of propionate breakdown. Accumulated propionate, in turn, leads to an inhibition of acetate utilizing methanogens.

c. <u>Influence of Detention Time</u>: The effect of solids residence time, in general, has been explained in an earlier section. In completely mixed systems, all the components of the digesting fluid have the same residence time in the system. Therefore, the solids retention time is equal to the liquid detention time.

It has been reported by Taiganides [39] that stable anaerobic digestion of swine waste in completely mixed systems can be sustained with a detention time of eight days at 35° C. However, a minimum of ten days is recommended to improve volatile solids reduction.

The findings of Dague [12] have been corroborated by researchers experimenting with anaerobic treatment of swine waste. It has ben reported by Van Velsen [56] that methane production is strongly affected by detention time between 10 and 15 days at 30° C days. A steep decline in the methane production is observed on reducing the digestion temperature from 35° C to 30° C. Summers

and Bousfield [55] experimented with seeded reactors fed swine waste containing 2% total solids. A rapid decline in gas production, solids reduction, COD reduction, and rapid increase in volatile acid concentrations has been reported at detention times less than 10 days at 35° C. Complete failure has been reported below a detention time of 3 to 5 days.

.

## III. EXPERIMENTAL SETUP

The experimental setup consisted of three identical, completely mixed reactors. The reactors were mixed by recirculation of the head space gas through the reactor contents. Figure 3 is a schematic of a typical setup for each reactor.

## A. Reactor Configuration and Design

The typical construction of a reactor is shown in Figure 4. The reactors were made from a 12 mm.(0.5 in.) thick PLEXIGLAS<sup>1</sup> tube. Each reactor had an inside diameter (I. D.) of 127 mm.(5 in.), outside diameter (O. D.) of 152 mm.(6 in.), and a length of 912 mm.(36 in.). The total reactor volume was 14 liters. The reactors were operated with an active volume of 12 liters. The reactors were calibrated at 0.5 liter increments throughout their length.

The reactors had circular flanged ends at the top and bottom. Each flange consisted of two circular, PLEXIGLAS plates each 12 mm.(0.5 in.) thick and 228.6 mm.(9 in.) in diameter. Flush with each end of the reactor was glued a plate, with a circular opening 152 mm.(6 in.) in diameter to exactly match the O. D. of the reactor. This plate, as well as the other one bolted to it, were provided with matching semicircular grooves to place an 'O' ring rubber gasket. The two flange plates were securely fastened by a set of twelve, 10 mm.(3/8 in.) flange bolts placed around the circumference of the flanges at a pitch of 30° and a distance of 12 mm. from the edge .

In addition, the reactors were equipped with nine effluent decant ports, with the first one placed 50 mm.(2 in.) from the reactor top and the rest spaced at 102 mm.(4 in.) along the length. The effluent ports were 25 mm.(1 in.) lengths of 16 mm.(5/8 in.) O. D. PLEXIGLAS tubes that were glued flush with the inner surface of

<sup>1</sup> Trade mark



Figure 3. Schematic of experimental setup



Figure 4. Schematic of reactor construction

the reactor tube. One hundred mm. lengths of 10 mm.(3/8 in) I. D. TYGON<sup>2</sup> tubing were fitted to each of the effluent decant ports and were clamped during normal operation of the reactor.

A set of four tubes was mounted on the top flange of each reactor. Two of the tubes (16 mm.(5/8 in) in diameter, 38 mm.(1.5 in.) in length), and made of PLEXIGLAS, were mounted on the top flange of each reactor. These tubes served as gas exit points to the gas handling system, as shown in Figure 4. One of the other tubes was a 12 mm.(0.5 in.) diameter stainless tube that extended to the reactor bottom and had a porous plate ceramic diffuser attached to it after a right angle bend. This tube was fitted to the top flange using a compression fitting and was used for the return of recirculation gas. The other tube, 25 mm.(1 in.) in diameter and also of stainless steel, extended to 152 mm.(6 in.) above the reactor bottom and was used to feed the reactors.

The three reactors were housed in a constant temperature incubator. The incubator was a Fischer Isotemp incubator capable of maintaining temperatures in the range of  $-10^{\circ}$  C to  $+50^{\circ}$  C with an accuracy of  $+/-0.2^{\circ}$  C. The rest of the equipment was kept outside of the incubator at room temperature ( $25 \pm 3^{\circ}$  C).

# B. Gas Handling System

The gas exiting the reactor was conveyed to the gas handling system. The gas piping in the system was built using 3/8 in.(10 mm.) I. D. x 1/16 in. (1.5 mm.) thick TYGON tubing. The gas exited the reactor into a foam separation bottle placed to intercept solids carried by the gas. Removal of the foam and particulates from the recirculation gas was essential to prevent clogging of the fine pores of the

<sup>&</sup>lt;sup>2</sup> Trade mark

gas diffuser. The foam separation bottle was a 4 liter aspirator bottle with a spout at the bottom. The bottle's rubber stopper had three 10 mm.(3/8 in.) tubes. One tube extended to the bottom of the bottle and the other two were short tubes. The long tube and the spout of the bottle were connected to the head space gas ports. Of the other two tubes in the stopper, one was connected to the suction end of the recirculation pump and the other to the water lock. Suction created by the pump caused the head space gas to be drawn to the pump through the bottle. The gas entered the bottle at its bottom and travelled upward towards the pump's suction end. Any foam or particles, carried by the gas, settled in the bottle yielding clean gas for recirculation.

The gas recirculation system consisted of a 6 to 600 rpm perstaltic pump and the porous plate diffuser at the bottom of each reactor. A 8 mm.(0.31 in.) I. D. neoprene tube was used in the pump. The pump was capable of pumping fluids at flow rates of 22.8 to 2280 mL. per minute at a pressure of 20 psi. The diffuser was a bubble disk, 51 mm.(2 in.) in diameter, which is commonly used as an aerator in small aquariums. A Chronotrol, four channel, 10 program, timer was used to turn the gas recirculation/ mixing on and off at particular time(s) of the day imposing the various phases of sequencing on the system. All of the three gas recirculation pumps were connected to a single timer.

The water lock consisted of a pair of interconnected columns filled with acidified water (pH= 2.0). The water lock allowed effluent removal without drawing air into the system as well as maintain a constant positive pressure on the system. Both the columns were fabricated of a PLEXIGLAS tube, 152 mm.(6 in.) O. D. x 127 mm.(5 in.) I. D and 292 mm.(11<sup>1</sup>/<sub>2</sub> in.) long. A circular plate, 152 mm.(6 in.) in diameter and 12 mm.(<sup>1</sup>/<sub>2</sub> in.) thick was glued on to each of the tubes and formed

the top cover. The bottom of each of the two columns was formed by a 203 mm.(8 in.) square and 12 mm.(1/2 in.) thick, PLEXIGLAS plate.

Of the two, the column marked A in Figure 5 was connected to the reactor through the foam separation bottle. The top cover had three, 16 mm.(5/8 in.) O. D. x 10 mm.(3/8 in) I. D., tubes glued on to it. The shortest tube (labelled #1), only 38 mm.( $1^{1}/_{2}$  in.) long, was glued at the center of the top cover, flush with the lower side of the cover. This tube was connected to the foam separation bottle. The other two were placed on either side of this tube, 51 mm.(2 in.) from its center. The longest one (labelled #2), 330 mm.(13 in.) long, extended to the bottom of the column, and was connected to the other column in the water lock. The third tube (labelled #3), had an intermediate length of 292 mm.( $11^{1}/_{2}$  in.) long and had a slanted lower end. It was used to release excess gas periodically through a sulfide scrubber, to the gas meter and the building's ventilation system.

The other column comprising the water lock was similar in construction and has been marked Column B in Figure 5. This column had two, 16 mm.(5/8 in.) O. D. x 10 mm.(3/8 in.) I. D., tubes glued on to the top cover. The short one, was again 38 mm.( $1^{1}/_{2}$  in.) long and was on to the top cover, flush with its lower side. The longer one was 330 mm.(13 in.) long, and was glued with the end reaching the bottom of the column, like in column A. This tube was connected to the longest tube in column A using TYGON tubing and a siphon was established between the two columns. The short tube was left open to the atmosphere.

The excess gas released from the reactor flowed into column A, displacing an equivalent volume of water into column B, through the interconnecting tube. The liquid level in column A was thus lowered and that in B raised, exerting increasing



Figure 5. Schematic of water lock displacement columns

pressure on the system. This process continued until the level in column A dropped below the lower end of the tube #3 and gas was released to the meter. Following release of the gas, the pressure would drop and the level in column A would rise above the end of tube #3.

The gas exiting from the water lock was cleaned using an iron oxide scrubber. The gas was first bubbled through acidified water in an observation bottle. Both the observation bottle and the scrubbers were made using one liter, glass stopper bottles. The glass stoppers were replaced by rubber stoppers. Two 3/8" O. D. glass tubes were placed in the stopper, one long and extending to the bottom of the bottle and the other short and extending just beyond the lower end of the rubber stopper.

The observation bottled was half filled with water acidfied to a pH of 2. Absorbent plastic foam pieces, soaked in a colloidal ferric oxide suspension were used to fill the scrubber bottle. The foam pieces were dried, after being soaked in a colloidal suspension of ferric oxide, before use. The gas would first enter the observation bottle and then the scrubber. The hydrogen sulfide in the gas reacted with the ferric oxide scrubber forming ferric sulfide and was thus removed. Scrubbing the gas clean of sulfide eliminated corrosion of the ferrous parts in the gas meters.

A gas sampling port was placed beween the scrubber and the gas meter to enable sampling of gas for chromatographic analysis. The sampler was fabricated out of a 25 mm.(1 in.) O.D. x 63 mm.( $2^{1}/_{2}$  in.) long glass tube. A 12 mm.( $^{1}/_{2}$  in.) length of a 10 mm.( $^{3}/_{8}$  in.) O. D. glass tube was provided at either end of the sampler for the connection of TYGON tubing. At the center of the sampler tube, a

16 mm.( $^{5}/_{16}$  in.) I. D. glass tube was provided. A rubber septum was inserted in this tube and served as the sampling point.

#### C. Gas Measurement

The gas produced in the reactors was measured using wet tip gas meters proccured from Rebel Wet Tip Gas Meter Company of Nashville, Tennessee. Details of the meter are shown in Figure 6. The meters work on the principle of buoyancy. The meters are filled with water and the gas is admitted at the bottom under a float. The float is hinged at mid length of the meter and has a stainless steel spherical weight. As the gas accumulates under the float, the float becomes increasingly buoyant. At a certain point, the buoyancy exceeds the weight of the sphere and the float tips about the pivot. The weight and travel of the sphere are so adjusted that accumulation of 100 mL. of gas causes a tip, with the float fully submerged. The float is so arranged that a single orifice is adequate to release gas under either side of the float. The tipping action releases the gas to a vent above the water surface, and causes the switching of ferrous contacts as well. The switching of ferrous contacts is achieved by a magnet attached to the float. The switching advances an electronic counter, connected to the contacts, by a count of one. Therefore, by counting the number of tips, the gas production can be measured. Given the volume of gas production expected from the reactors, this form of gas measurement is considered to be sufficiently accurate.



Figure 6. Schematic of the wet tip gas meter

#### IV. EXPERIMENTAL PROCEDURES

#### A. Reactor Feed Preparation

Swine waste, collected from Iowa State University's Swine Nutrition Farm, was used as a feed for the reactors during this study. The waste was collected from the finishing facilities that housed animals in weight range of 100 - 200 lbs. At this age, the composition of fecal matter excreted by the animals is fairly consistent. The waste collected was primarily fecal matter scraped from the floor of the confinement building. The waste was collected once a month and stored frozen in five gallon containers.

Prior to use as feed, the waste was diluted by factor of 4 and homogenized. A Waring blender was used to homogenize the waste. The jar of the blender was tared on a top loading balance and 1500 g. of waste was added. Water was added to the jar to take the volume to five liters. The waste was blended to a uniform consistency. The blended waste was refrigerated in two liter bottles until use. Dilution of the waste was essential to prevent inhibition from high ammonia levels. The characteristics of the raw manure and blended feed are listed in Table VII. The strength of the feed was periodically checked by measuring its chemical oxygen demand (COD) using the Closed Reflux method given in Standard Methods [61]. Since the method listed in the reference is capable of measuring COD only up to 340 mg/L, the feed samples were diluted by a factor of 250 prior to determination.

## B. Reactor Start-up

The reactors were operated with an active volume of 12 liters. The reactors were started with a total COD load of 1 g/L/d at  $35^{\circ}$  C. To start the reactors, 10 liters of actively digesting anaerobic sludge, blended feed corresponding to a total

COD of 12 grams (1 g/L x 12 liters), and tap water to take the total volume to 12 liters were added to each reactor. The incubator temperature was set at  $35^{\circ}$  C. Anaerobic digester sludge was used as a seed and was obtained from the primary digesters at the Ames, lowa, wastewater treatment plant. Prior to use, the seed sludge was sieved through a 1 mm screen to remove grit and other foreign matter.

Characteristic	Raw Manure	Blended Feed
Total Solids, %	18 - 26	5.3 - 7.2
Volatile Solids, %	15 - 24	4.6 -7.0
COD, g/L	210 - 340	64 -102
Five day BOD, g/L	73 - 80	22 - 24
COD/BOD <sub>5</sub> ratio	1.8 - 2.0	1.8 - 2.0
рН	4.7 - 5.8	6.2 - 6.4
Total Kjeldahl Nitrogen, g	g/L as N 13 - 15	3.8 - 4.6

Table VII. Characteristics of Raw Swine Manure and Blended Feed

After start-up, the reactors were not mixed for a period of approximately 24 hours in order to exhaust any oxygen present in the reactors. The reactors were operated at a total COD load of 1 g/L/d for a period of two weeks until equal gas production (+/- 2%) was achieved in all reactors.

# C. <u>Reactor Operation</u>

The reactors were sought to be operated at loads of 1, 2, 3, 4 & 5 g COD/ L/day at  $35^{\circ}$  C and  $25^{\circ}$  C, and a six day hydraulic retention time (HRT). The reactors were fed once per day and were operated in a sequencing batch mode. The lengths of the various phases of the sequencing cycle were as follows:

Settling Phase	2.0 Hours
Decant Phase	0.5 Hours
Feed Phase	0.5 Hours
React Phase	21.0 Hours

After the initial period of two weeks allowed for acclimation of the reactors, the reactors were operated in four phases.

In Phase I, the load on two of the reactors was changed to 3 g COD/L/d. The reactors were operated at 1 g/L/d (reactor #2) and 3 g/L/d (reactors #1 & #3) until the COD and solids removal efficiency, and daily methane production in each reactor had stabilized to a constant value. Data were collected in terms of the parameters and frequency listed in Table VIII.

Parameter	Monitoring Frequency
Gas Production	Daily
рН	Thrice a week
Gas Composition	Twice a week
Solids Retention Time	Weekly
Total Alkalinity	Weekly
Total Volatile Acids	Weekly
Solids Removal, %	Weekly
COD removal, %	Weekly

 Table VIII.
 Reactor Performance & Monitoring Parameters

In Phase II, the loads on the reactors were again increased while keeping the temperature constant at  $35^{\circ}$  C. The load on reactor #2, which was operating at 1 g/L/d, was changed to 2 g/L/d. The load on the other two reactors was changed to 4 g/L/d. After operating for a week, the load on reactor #1 was increased further to 5 g/L/d. Reactor operation was continued at these loads and system performance data were collected.

In Phase III, the temperature of reactor operation was changed from 35° C to 25° C. The temperature of the incubator was changed from 35° C to 25° C over a period of one hour. In order to avoid both load and temperature shocks, the reactors were not given feed on the day the temperature was changed. Reactor feeding was resumed the next day with reduced loads, 1 g/L/d (reactor #2), 2 g/L/d (reactor #3) and 3 g/L/d (reactor #1). Reactor operation was continued until the reactors attained stable performance values (+/- 2 %). In phase IV, the reactors were operated at loadings of 4 g/L/d (reactor #3) and 5 g/L/d (reactor #1).

#### D. Daily Reactor Maintenance Procedure

The reactors were batch-fed once per day. The daily reactor maintenance procedure comprised of the following steps:

1. <u>Recording the gas reading in the wet tip gas meters</u>.

In order to normalize the daily gas production to standard temperature and pressure conditions (760 mm. Hg & 273° K), the ambient pressure and temperature were measured at the time of recording the gas reading. The ambient pressure was measured using a mercury barometer. The barometer had an accuracy of +/- 0.1 mm. The temperature was measured using a mercury thermometer capable of measuring temperature in the range of -20° C to 100° C (+/- 0.1° C).

The gas meter readings, barometric pressure, and ambient temperature were used to calculate the daily gas production at the standard temperature and pressure (STP) of 0° C and 1 atmosphere by the following equation developed from the ideal gas law:

$$G = \left[\frac{\frac{p1+p2}{2}}{\frac{ps}{2}}\right] \left[\frac{273}{\frac{273}{2}(\frac{T1+T2}{2})}\right] \left(\frac{V2-V1}{t}\right)$$

where,

G = gas production rate at STP (L/d),

p1= barometric pressure at previous gas reading (mm. Hg),

p2= barometric pressure at current gas reading (mm. Hg),

Ps= standard pressure= 1atm = 760 mm. Hg,

T1= temperature at previous gas reading (° C),

T2= temperature at current gas reading (° C),

V1= previous gas reading (liters),

V2= current gas reading (liters) and

t = time elapsed between current and previous gas readings (days)

### 2. Decanting the supernatant at the end of the settle phase.

The reactors were operated with an active volume of 12 liters at an HRT of six days. Therefore, two liters of supernatant were wasted out of the reactors each day. The second and third (from reactor top) effluent ports were used for supernatant withdrawl.

## 3. Effluent Sample collection for parameter monitoring.

Grab samples for pH and alkalinity were collected while decanting the reactor with minimal exposure to the atmosphere to insure that no carbon dioxide was lost from the samples. Samples for pH were collected in a 10 mL sample bottle with a ground glass stopper and were capped. Samples for alkalinity were collected in 50 mL. Erlenmeyer flasks and immediately capped with a polyethylene film. The alkalinity and pH of the samples was determined immediately. On the

other hand, the effluent samples for COD, solids, and volatile acids determinations were collected after the composition of the entire two liter volume of the supernatant taken from each of the reactors. Composition of the entire effluent decanted was considered to be essential in order to eliminate variation in the supernatant at the various depths in the reactor.

### 4. Addition of blended feed corresponding to the COD load.

The reactor feeding tubes were cleaned to remove clogs etc, using a 12 mm.(1/2 in.) O.D. rigid polyethylene pipe. Blended feed, corresponding to the COD load being applied to each reactor, was added through the feed tube using a plastic funnel. The active volume of each reactor was made up to 12 liters, by the addition of tap water. The tap water also served to insure that the desired quantities of feed were introduced into the reactor and not left in the feed pipe. 5. Start of mixing and the react phase.

# E. Laboratory Analyses

# 1. <u>pH</u>

The pH in the reactors was monitored on a thrice per week basis. The pH measurements were made using an Altex Instruments Model 4500 digital pH meter (accuracy +/- 0.001 pH) and a Markson electrode. The pH meter was calibrated each time prior to use using a buffer solution of pH 7.0. Since the pH in the reactor was expected to be near 7.0, calibration with a buffer of pH 7.0 was considered adequate.

# 2. Total alkalinity and total volatile acids

Total alkalinity and total volatile acids were determined using the methods outlined in Standard Methods [61]. Total alkalinity was determined by the titration of a 25 mL. effluent sample to a pH of 4.3 using dilute (0.1 N) sulfuric acid. Typically, the total alkalinity is constituted by the bicarbonate alkalinity and alkalinity due to the volatile acids and other ions such as phosphate. The principal reactions occurring during the titration are:

$$2NH_4HCO_3 + H_2SO_4 -----> (NH_4)_2SO_4 + H_2O+ H_2CO_3$$
  
 $2CH_3COONH_4 -----> (NH_4)_2SO_4 + 2CH_3COOH$   
 $2(NH_4)H_2PO_4 -----> (NH_4)_2SO_4 + 2H_3PO_4$ 

The total alkalinity was calculated using the following expression given in Std. Methods:

where

N = normality of sulfuric acid.

mL.  $H_2SO_4$  = volume of sulfuric acid used in titration to pH of 4.3

Total volatile acids in the effluent were determined by the distillation method. The effluent was centrifuged at 1000xg to remove the settleable solids. A 100 mL. aliquot of the supernatant was taken and distilled with 100 mL. of distilled water and 5 mL. of concentrated sulfuric acid (39 N). After discarding the first 5 mL., 150 mL. of the distillate were collected. The collected distillate was titrated with dilute sodium hydroxide (0.1 N) to a pH of 8.3. The total volatile acids were calculated as:

where, mL. NaOH = volume of sodium hydroxide used in titration to pH 8.3 N = normality of sodium hydroxide titrant

# 3. Gas analysis

The gas produced by the reactors was analyzed twice per week for the relative proportions of  $CO_2$ ,  $CH_4$  and  $N_2$  by using a gas chromatograph (GC) with

a thermal conductivity detector. The operating conditions for the GC used are presented in Table IX.

Gas Chromatograph	Hewlet Packard 5730A
Column	6 ft. x 3 mm I.D., stainless steel
Packing	Poropak Q, 80/100 mesh
Temperature	Ambient
Carrier gas	Helium
Flow rate	30 mL. / min.
Column pressure	60 psig
Detector	Thermal conductivity
Temperature	200° C
Bridge current	225 mA
Sensitivity	10 mA
Injection point temperature	100° C
Sample size	0.9 mL.

Table IX. GC operating conditions for gas analysis.

A standard was used to establish the calibration curve. The standard consisted of 70%  $CH_4$ , 25%  $CO_2$  and 5%  $N_2$  (concentrations within +/- 0.5 %). Four samples of standards were used, two in the beginning of the sample queue and two at the end. The gas samples from the reactors were taken from the gas samplers, equipped with silicone rubber septa, located between the scrubbers and the gas meters. The samples were taken with HAMILTON<sup>1</sup> Gas tight #1001TLL

<sup>1</sup>Trade mark

syringe. The syringe was pre-treated by withdrawing and discarding three samples before taking a sample for analysis. The sample volume drawn was larger than 0.9 mL., and was adjusted to 0.9 mL. just before injection. Duplicate samples were run for each reactor. The peak areas of the standards were used to calculate the response factor. Peak identification and integration was done using a Maxima data station.

## 4. Chemical oxygen demand

The COD test was performed once per week on the mixed liquor and effluent samples from the reactors as well as the stock feed solution. It was used as a measure of the oxygen equivalent of the organic material in the samples that could be oxidized by the strong chemical oxidant, potassium dichromate, in an acid solution. The CODs were analyzed using the Closed Reflux Titrimetric Method (method # 508 B) [61]. The digestion vessels used were 20 x 150 mm. borosilicate culture tubes with PTFE lined phenolic screw caps. The sample and reagent quantities used were as follows:

Sample	= 5.0 mL.
Potassium dichromate digestion solution	= 3.0 mL.
Sulfuric acid reagent	= <u>7.0 mL.</u>
Total	= 15.0 mL.

Because this variation of the COD test has a theoretical COD measuring capacity of 480 mg  $O_2/L$ , it was necessary to dilute all of the samples appropriately. For the effluent and stock feed samples, both a "total" COD (TCOD) and a "soluble" COD (SCOD) were determined. The TCOD was determined on the sample as it came from the reactors or stock feed bottle and diluted appropriately with distilled water. Since the samples from the reactors and the feed contained

coarse particulates, the samples were always stirred during dilution and collection of COD aliquots.

The SCOD was determined on the filtrate samples resulting from centrifugation (@1000 x g) and vacuum filtration through a Whatman GF/C glass fiber filter disk. The filtrate was then diluted as needed with distilled water.

All samples and blanks used in the COD determination were run in duplicate. The COD was calculated as follows:

where

A = mL. FAS titrant used for blank, B = mL. FAS used for the sample, and M = molarity of FAS titrant

# 5. <u>Solids</u>

Solids analyses were performed on the mixed liquor and effluent samples as well as samples of the stock feed. The samples were analyzed in duplicate for total suspended solids as per Standard Method 209C [61] and for volatile suspended solids according to Standard Method 209 D. The sample sizes were 10 mL. for the samples from the reactors and 3 mL. for the stock feed samples. The glass fiber filter disks used were Whatman GF/C with a diameter of 9 cm. Large aluminium planchets were used to support the filters, and the filters and planchets were weighed together.

The following modifications were made on Standard Method 209 C for total solids analysis:

a. The filter disks were prepared by igniting at 610 +/- 10° C in a muffle furnace for 15 minutes and were then cooled in a dessicator. They were then weighed immediately before use.

b. After filtering the sample, the filter was washed twice with 10 mL. of distilled water.

c. After the filtration was complete, the filters were dried, cooled, dessicated and weighed once.

The total suspended solids were calculated as follows:

where

A = weight of filter + planchet + dried residue, g; and

B = weight of filter + planchet, g.

Standard method 209D for fixed and volatile solids was modified by only igniting, cooling, desiccating and weighing the filters once. The volatile solids were calculated as follows:

Volatile Suspended Solids = (A - B)(1000 mg / g)(1000mL./L) sample volume, mL.

where,

A = weight of filter + residue + planchet before ignition, g, and

B = weight of filter + residue + planchet after ignition, g.

The solids (suspended + dissolved) content was estimated using the following relation:

## 6. <u>Ammonia</u>

Ammonia analyses were performed weekly on the effluent samples from the reactors and on the stock feed solution. The ammonia determinations were made by following Standard Method 417E, the ammonia-selective electrode method [61]. The method was modified by using 50 mL. volumes of the standards and samples instead of the recommended 100 mL. The samples were not diluted.

The ammonia -selective electrode used was an Orion Research Model, 95-12. The electrode was used in conjunction with a Altex Instruments Model 4500 digital pH meter. A semi-log calibration plot of millivolts vs log (concentrations) was developed using the standards. The  $NH_3$ -N concentration was calculated from the calibration curve.

#### 7. <u>Total Kieldahl Nitrogen</u>

The Total Kjeldahl Nitrogen content of the waste and blended samples was determined periodically using the Block Digester - Potentiometric method [62]. The samples were digested with an acidic solution of potassium sulfate with mercuric ion as a catalyst. The organic nitrogen in the sample is reduced to ammonium ions. The concentration of the ammonium ion was measured using the potentiometric method described earlier. The procedure is outlined below:

a. A 20 mL., adequately diluted, sample was placed in a MICRO KJELDAHL digestion tube (25 mm. I.D. x 229 mm. long).

b. 5mL. of digestion solution and a few TEFLON boiling chips were added to the sample.

c. The samples were digested in a TECHNICON block digester, first at 200° C for 60 minutes and then at 380° C for 90 minutes.

d. Following digestion and cooling, the samples were diluted by the addition of 25 mL. of distilled and deionized water.

e. The nitrogen content was measured as ammonia using an ammonia selective electrode.

.

## V. RESULTS AND DISCUSSION

The performance of the reactors was monitored at regular intervals. Quasisteady state data was used to quantify reactor performance. The reactors were considered to be at quasi-steady state when the daily methane production was fairly constant (+/- 2%) and consistent COD removals were being achieved. The following sections summarize the quasi-steady state data obtained during the study. The data collected during regular monitoring is presented in Appendices A through E.

## A. Influence of Organic Loading Rate

The goal of this study was to operate the reactors at organic loads of 1, 2, 3, 4 and 5 g COD/L/day. The actual loads achieved varied slightly from the targets. The actual loads achieved are listed in Table X.

Temperat Total COD Load, g/L/day	ure = 35º C /olatile Solids Load, g/L/d	Temperatur Total COD \ Load, g/L/d	e = 25º C /olatile Solids Load, g/L/d
1.005	1.09	1.140	1.04
2.167	2.24	2.280	2.09
3.283	3.26	3.420	3.13
4.372	4.44	5.000	6.03
5.436	5.38	5.670	6.82

Table X. Total COD and Volatile Solids Loadings at 35° C & 25° C

The system performance was evaluated in terms of gas production and the organic removal efficiencies. The removal efficiency was calculated using the following relationship:



- $S_i$  = Influent organics (COD or volatile solids) / day
- $S_0 =$  Effluent organics (COD or volatile solids) / day
- M = average algebraic daily change in the total organic content (COD or volatile solids) of the reactor

The average COD removal and gas production data obtained at the various COD loading rates evaluated are shown in Tables XI and XII. The volatile solids removal data obtained at the various COD loading rates are shown in Tables XIII and XIV.

The removal efficiency was found to decline with increase in load. This behavior is clearly evident in the plots of COD removal versus COD load (Figure 7) and, Volatile Solids (VS) removal versus VS load (Figure 8). The removal efficiency is seen to decline from about 80% at a load of 1 g /L /day to about 62% to 63% at 3 g/L/day at both the temperatures investigated. The decline is due to an increase in effluent solids content between the two loads. At 35° C, the effluent volatile solids concentration was only 440 mg/L at a load of 1 g/L/day. It rose to over 4700 mg/L at a load of 3 g/L/day (Tables XIII & XIV). The settling phase at 1 g/L/d was characterized by a zone of clear supernatant overlying a zone of dense solids. The supernatant became increasingly turbid with the increase in load.

The variation in removal efficiency with load is reflected in the variation of gas production with load. The variation of gas production is shown in Figure 9. It is seen that the slope of the curves changes with increase in load. The slope change is rapid between loads of 1 and 3 g/L/day and gradual beyond 3 g/L/d. In this

COD Load g/L/day	Influent COD g/day	Effluent COD g/day	M (COD) g/day <sup>a</sup>	COD Removal %	Gas Production L/day, STP	CH4 %
1.005	12.06	2.60	0	78.4	3.99	71.4
2.167	26.00	7.40	0	71.5	11.30	62.1
3.283	39.40	13.02	0	67.0	16.50	62.7
4.372	52.46	17.82	+0.49	65.1	20.20	61.5
5.436	65.24	20.34	+2.79	64.6	30.30	62.6

Table XI. COD and gas production performance data at 35° C

<sup>a</sup> The M(COD) column indicates the average daily increase (+) or decrease (-) in the COD of the mixed liquor during the run for each COD load. This change in mixed liquor inventory is accounted for in the calculation of the COD removal percentage.

COD Load g/L/day	Influent COD g/day	Effluent COD g/day	M (COD) g/day <sup>a</sup>	COD Removal %	Gas Production L/day, STP	CH4 %
1.14	13.70	4.88	-2.424	82.1	4.95	66.5
2.28	27.36	6.04	0	77.9	12.52	63.1
3.42	41.04	12.20	+3.06	62.8	17.55	61.3
5.00	60.10	23.14	-2.46	65.6	22.82	58.1
5.67	68.00	25.36	-1.58	65.0	26.97	56.0

Table XII. COD and gas production performance data at 25° C

<sup>a</sup> The M(COD) column indicates the average daily increase (+) or decrease (-) in the COD of the mixed liquor during the run for each COD load. This change in mixed liquor inventory is accounted for in the calculation of the COD removal percentage.

COD Load g/L/day	VS Load g/L/day	Influent VS g/day	Effluent VS g/day	M (VS) g/day <sup>a</sup>	VS Destruction %
1.005	1.09	13.08	0.88	+0.759	87.4
2.167	2.24	26.88	4.28	+1.418	78.8
3.283	2.75	39.20	9.44	-0.501	77.2
4.372	4.44	53.28	13.84	0	74.0
5.436	5.38	64.56	16.08	+0.960	73.6

Table XIII. Volatile solids performance data at 35° C

<sup>a</sup> The M(VS) column indicates the average daily increase (+) or decrease (-) in the VS of the mixed liquor during the run for each COD and VS load. This change in mixed liquor inventory is accounted for in calculating the VS removal percentage.

COD	VS	Influent	Effluent	M	VS
Load	Load	VS	VS	(VS)	Destruction
g/L/day	g/L/day	g/day	g/day	g/day <sup>a</sup>	%
1.140	1.04	12.48	0.95	+0.097	91.7
2.280	2.09	25.08	4.98	+0.369	78.7
3.420	3.13	37.56	10.74	-2.820	78.9
5.000	6.03	72.36	16.10	0	77.7
5.670	6.82	81.84	18.92	0	76.9

Table XIV. Volatile solids performance data at 25° C

<sup>a</sup> The M(VS) column indicates the average daily increase (+) or decrease (-) in the VS of the mixed liquor during the run for each COD and VS load. This change in mixed liquor inventory is accounted for in calculating the VS removal percentage.



Figure 7. Reduction in total COD at temperatures of 25° C and 35° C.



Figure 8. Volatile solids destruction at temperatures of 25° C and 35° C.

COD Load	VS Load	SRT	рН	Alk <sup>a</sup>	VA <sup>b</sup>	NH <sub>3</sub> c	MLSS <sup>d</sup>
g/L/day	g/L/day	days		mg/L	mg/L	mg/L	mg/L
1.005	1.09	107	6.7	1080	8	240	7390
2.167	2.24	20	6.8	1458	16	307	11220
3.283	2.75	17	6.9	2580	24	398	10950
4.370	4.44	13	7.1	2443	27	588	18950
5.436	5.38	13	7.1	2896	31	822	18350

Table XV. SRT and system parameters at 35° C

<sup>a</sup> Total alkalinity, as CaCO<sub>3</sub>

<sup>b</sup> Total volatile acids, as CH<sub>3</sub>COOH

<sup>C</sup> Ammonia, as N <sup>d</sup> Mixed liquor suspended solids

COD Load	VS Load	SRT	pН	Alk <sup>a</sup>	VA <sup>b</sup>	NH <sub>3</sub> c	MLSS <sup>d</sup>
g/L/day	g/L/day	days		mg/L	mg/L	mg/L	mg/L
1.140	1.04	125	7.0	2535	15	138	10270
2.280	2.09	41	7.1	3393	28	290	17910
3.420	3.13	21	7.1	4232	74	463	20360
5.000	6.03	22	6.8	2840	40	449	24050
5.670	6.82	20	6.8	3160	160	600	29670

Table XVI. SRT and system parameters at 25° C

<sup>a</sup> Total alkalinity, as CaCO<sub>3</sub>

<sup>b</sup> Total volatile acids, as CH<sub>3</sub>COOH

<sup>C</sup> Ammonia, as N <sup>d</sup> Mixed liquor suspended solids
study, the highest loads investigated were about 5 g/L/d. Further experimentation is needed to ascertain the system performance at higher loads.

The system chemical parameters such as ammonia, pH, alkalinity and volatile acids increased with increase in load. At higher loads, the system was characterized by higher ammonia concentrations. This probably led to the formation of increased amounts of ammonium bicarbonate in the system resulting in higher alkalinity and pH. The methane content (%) of the gas decreased as increasing amounts of carbon dioxide were released from the liquid to the gaseous phase at the higher loads. The chemical parameters are listed in Tables XV and XVI.

It is interesting to compare the performance of the ASBR with conventional continuously mixed digesters treating swine waste. Results from this study are compared, in Figure 10, with those reported by three other researchers using completely mixed reactors [47,54,56]. The work of Fischer, et al. [54] and, Hobson and Shaw [47] was conducted at  $35^{\circ}$  C. The studies by Van Velsen [56] were conducted at  $30^{\circ}$  C (+/-  $2^{\circ}$  C). As shown in Figure 10, volatile solids destruction at equivalent loads in this research (Pidaparti) are much higher than the results reported by Hobson and Shaw and Van Velsen and Fischer et al. This indicates that the ASBR is much superior in performance to conventional continuously mixed digesters.

#### B. Influence of Temperature

Temperature has a significant influence on the rates of synthesis, metabolism and decay in microorganisms. In a system containing all the necessary nutrients and optimal environmental conditions for growth, the most important factor affecting

69



Figure 9. Gas production at temperatures of 25° C and 35° C.



Figure 10. Comparison of performance - ASBR versus conventional CSTRs

the rate of microbial growth is temperature [12]. Over normal temperature ranges, growth rates decrease with decreasing temperature and increase with increasing temperature [12]. Microbial rates usually double for every 10° C rise in temperature and decrease by half for every 10° C drop in temperature.

In this study, the reactors were first operated at 35° C (phases I & II). In phases III & IV, the temperature was dropped to 25° C and the reactor operation was attempted at the same loads as in phases I & II. The quasi-steady state data collected at the two temperatures is presented in Tables X through XVI. The organic removal and gas production data is graphically represented in Figures 7, 8 and 9.

A drop in organics removal efficiency was expected on reducing the temperature from 35° C to 25° C. However, no significant change in the performance was observed. The gas production and removal efficiencies remained essentially unchanged. The various curves obtained at the two temperatures are almost identical. The curves indicate that the performance of the ASBR in treating swine waste was independent of temperature between 35° C and 25° C over the range of organic loads evaluated.

It has been suggested by Dague et al. [13] that solids retention, defined in terms of SRT, is the single important factor for equivalent removal efficiency and performance at low or high temperatures. It has been suggested that effects of temperature can be counteracted by altering microorganism population dynamics. In other words, a drop in temperature can be counteracted by a proportional increase in microbial numbers. The key to achieving equivalent degrees of treatment is the achievement of SRTs that are sufficiently longer at the lower temperature to compensate for lower metabolic rates. Figure 11 illustrates that the ASBR achieved longer SRTs at  $25^{\circ}$  C than at  $35^{\circ}$  C. At a COD load of 2 g/L/d, the SRT at  $25^{\circ}$  C was approximately twice the SRT at  $35^{\circ}$  C (40 days vs 20 days). Therefore the system was able to hold larger microbial populations and offset the reduction in temperature.

Higher SRTs are achieved at the lower temperature as result of the lower endogenous decay rates and the ability of ASBR to retain solids. The endogenous decay rates have been estimated based on the Modified Model presented by Dague for biological growth [63].

$$M_a = M_o \exp(-k_e * SRT)$$

where,

 $M_a$  = active mass,  $M_o$  = theoretical yield, and  $k_e$  = endogenous decay rate

Assuming the mixed liquor volatile suspended solids (MLVSS) to be a measure of the active mass, the decay rates at the two temperatures were estimated from plots of log (MLVSS) versus SRT (Figures 12 & 13). It is observed that the decay rate constant at 25° C (= 0.02 d<sup>-1</sup>) is substantially lower than the decay rate at 35° C (= 0.06 d<sup>-1</sup>). However, the theoretical biomass yield (the Y-intercept) is constant. The decrease in the decay rates is more than two fold and does not reflect the Q<sub>10</sub> trend suggested by Arrhenius law. It must be recognized that the decay rates have been evaluated on the basis of MLVSS. In any biological system, the MLVSS is only an approximate and not an accurate measure of the active biomass.



Figure 11. Solid retention times achieved at various total COD loads



SOLIDS RETENTION TIME, DAYS

Figure 12. Log(MLVSS) - SRT plot for 35° C



Figure 13. Log(MLVSS) - SRT plot for 25° C

#### C. Stability of the Effluent

One of the important criteria in swine waste stabilization is odor reduction. It is essential that the effluent, gases and sludge generated from the treatment process are devoid of odors and incapable of generating odors on storage. In order to assess the potential for generation of odors on storage, a one liter sample of effluent was incubated in a sealed Erlenmeyer flask at 35° C for a period of 22 days. The sample was taken from a reactor being operated at a COD load of 3 g/L/d and 25° C. Since volatile acid production is the principal source of odors, the volatile acids content in the sample was determined periodically.

The sample was devoid of odorous acids such as propionate, butyrate and valerate from the start and there was no significant increase of these compounds observed during the incubation period. At the end of the incubation, the sample had no persistent odors. This study indicated that the effluent from ASBRs treating swine waste is well stabilized and incapable of odor generation, even on storage at elevated temperature. Table XVII shows the development of volatile acids in the sample.

Date	Volatile Acids, ppm
2/21/91	16.9
2/28/91	15.1
3/15/91	52.8
3/21/91	25.4

Table XVII. Development of volatile acids in the incubated effluent sample

#### D. Shutdown Recovery Characteristics

Waste treatment systems located at farms or industries are often subject to periods of shutdown when no influent waste is available. Recovery of the system following such periods, without seeding or restart, is a desirable characteristic in such waste treatment systems.





Figure 14. Recovery of gas production following shutdown

During phase IV, the reactor feed for this study was unavailable for about a week following relocation of the the Swine Nutrition Farm. As a result, the reactors were shut down for a week and started at full load after the feed was available. The recovery of gas production in the ASBR after start-up, at a COD loading rate of 5.7 g/L/day and 25° C, is shown in Figure 14. It was observed that the ASBR

completely recovered in about 100 hours, even at the high load and low temperature conditions.

#### E. Engineering Considerations

#### 1. <u>Sizing of a full scale system</u>

The economics of waste treatment are controlled by the size of the waste treatment reactor and thus the highest loading rate that can be applied to the system. The smaller the loading rate, the larger the reactor and the higher the costs of installation, operation, etc. This section presents an estimation of the size of a full scale ASBR treating swine waste.

The size has been estimated using an average animal weight of 100 lb. Based on the data summarized by Dague [42], it is estimated the daily waste production of a 100 lb hog has a COD equivalent of 1.25 lb (567 g). Using an optimun COD loading of 5 gram per day per unit reactor volume,

ASBR volume required for 1000 hogs=  $\underline{113400 \text{ liters}} = \underline{4000 \text{ Cu. ft}}$ . (approx.) Therefore, the reactor dimensions are  $\underline{20 \text{ ft}}$ . x  $\underline{20 \text{ ft}}$ . x  $\underline{10 \text{ ft}}$ . for a 1000 hog confinement feeding facility.

#### 2. <u>Scheme for a treatment unit</u>

In order to minimize costs of construction, the ASBR system can be formed as a covered lagoon with gas mixing diffusers at the bottom. To mix the ASBR, the head space gas can be recirculated through the diffusers periodically. A small, secondary, open lagoon can be provided following the ASBR in order to retain bacterial solids and to allow for dissipation of sulfide odor. The secondary lagoon need not be mixed.

The supernatant from the secondary lagoon is aerated to remove any remaining sulfide odors and reused to flush the waste from the confinement facility. The suggested flow scheme is illustrated in Figure 15. Reuse of the effluent for flushing is possible as the effluent is fairly stable and does not pose problems of odor. However, due to the accumulation of cations in the effluent, the recycle needs to be diluted with an amount of make-up water that will prevent cation inhibition in the ASBR.



Figure 15. Flow scheme for swine waste treatment using the ASBR.

#### **VI. CONCLUSIONS**

The following conclusions can be drawn from this study:

1. The anaerobic sequencing batch reactor (ASBR) shows a significantly higher efficiency than conventional mixed digesters in treating swine waste. The removal efficiencies vary from 60% at a COD load of 5 g/L/d to 80% at a COD load of 1 g/L/d.

2. The ASBR can withstand temperature reductions between 35° C and 25° C without significant loss in efficiency. Therefore, the ASBR is particularly suited to the treatment of swine wastes in the temperate climate of the midwestern United States, where temperature swings are common.

3. The effluent from the reactor is odorless and incapable of producing odor on storage.

4. The reactor can recover quickly following shutdown.

#### VII. RECOMMENDATIONS FOR FURTHER RESEARCH

Based on the results of this study, several recommendations can be made with regard to conducting further studies on the ASBR and its applications to the treatment of swine waste. These are:

1. The experiments in this study have been conducted at a six day hydraulic retention time. Most swine confinement feeding facilities employ hydraulic flushing for removal of wastes. In such situations, where large volumes of water have to be handled by the system, the average flow rate or the HRT is primary factor in reactor sizing. Studies to determine the efficiency of the ASBR at lower HRTs are recommended.

2. This study has focussed on the operation of the ASBR at 35° C and 25° C. The efficiency of the ASBR at lower (psycrophillic) and higher (thermophillic) temperatures needs to be investigated.

3. As indicated in the literature review, there exists a potential for separate disposal for screened wastes and screenings. Study of the applicability of an ASBR to screened waste, is recommended.

4. In this study an attempt has been to determine the rate constant for decay at the two temperatures. The rate constant needs to be evaluated using a more rational basis for active biomass.

82

#### BIBLIOGRAPHY

- 1. The Des Moines Register, Des Moines, Iowa, "Iowa's Odor Problem." Editorial, November 30, 1988.
- 2. Taiganides, E. P. and Hazen, T.E., "Properties of Farm Animal Excreta." *Transactions of the American Society of Agricultural Engineers*, 9: 374 - 376, 1966.
- 3. Muehlling, A. J., "Swine Housing and Management, A Research Review." *Publication No. A Eng-873, College of Agriculture Cooperative Extension Service, University of Illinois, Urbana, Illinois*, 65-78, 1969.
- 4. Donham, K.J., Rubino, M.J., Thedell, T.D. & Kammermeyer, J., "Potential Hazards to Agricultural Workers in Swine Confinement Buildings." *Journal of Occupational Medicine*, 19: 383 -387, 1977.
- 5. Johnson, D., "Summary Information About Livestock Pit Fatalities." *Cooperative Extension Service, North Dakota State University of Agriculture and applied Science, Fargo, North Dakota*, Unpublished report, 4, 1981.
- 6. Donham, K.J. & Gustafson, K.E., "Human Occupational Hazards From Swine Confinement." *Annals, American Conference of Governmental Industrial Hygenists*, 2:137-142, 1982.
- 7. Miller, T.G., Living In The Environment: Concepts, Problems, Alternatives.
  Wadsworth Co., Belmont, California, 108-128, 1975.
- 8. Spoelstra, S.F., "Origin of objectionable odorous compounds in piggery wastes and the possibility of applying indicator components for studying odor development." *Agriculture and Environment*, 5: 241-260, 1980.
  - 9. Taiganides, E. P., Baumann, E. R., Hazen, T. E., "Anaerobic digestion of hog wastes." J. Agricultural Engineering Research, 8: 327-333, 1963.
- 10. Norman, J., "Depollution of Pig Waste Combined With Energy Recovery." Global Bioconversions. Donald L. Wise (ed.). CRC Press, Inc., Boca Raton, 1987.
- 11. Speece R. E., and McCarty, P. L., "Nutrient requirements and biological solids accumulation in anaerobic digestion." Proceedings of the First International Conference on Water Pollution Research, London, 1962.
  - 12. Dague, R. R., McKinney, R. E. and Pfeffer, J. T., "Anaerobic activated sludge." J. Water Pollution Control Federation, 38(2): 220 - 226, 1966.

- Dague, R. R., McKinney, R. E. and Pfeffer, J. T., "Solids retention in anaerobic waste treatment systems." *J. Water Pollution Control Federation*, 42(2): R29 -R46, 1970.
- 14. Bryant, M.P., "Microbial methane production theoretical aspects." *Journal of Animal Science*, 48(1): 193-201, 1979.
- Zeikus, J. G., "Microbial populations in digesters." *Anaerobic Digestion*. D.A. Stafford, B.I. Wheately and D. E. Hughes (eds.). Applied Science Publishers, Ltd., London, 1980.
- Wolfe, R. S., and Higgins, I. J., "Microbial biochemistry of methane a study in contrasts." *Microbial Biochemistry*. J. R. Quayle (ed.). University Park Press, Baltimore, 1979.
- 17. Daniels, L., "Biological methanogenesis: Physiological and practical aspects." *Trends in Biotechnology*, 2(4): 98-108, 1984.
- McInerney, M. M., Bryant, M. P., Hespell, R. B. and Costetron, J. W., "Syntrophomonas wolfei gen. nov sp. nov., an anaerobic, syntrophic, fatty acid oxidizing bacterium." Applied and Environmental Microbiology, 41(4): 1029-1039, 1981.
- 19. Daniels, L., Sparling, R. and Sprott, G.D., "The bioenergetics of methanogenesis." *Biochemica et. Biophysica Acta*, 768(2) 113-163, 1984.
- 20. Seagren, E. A., "High pH inhibition in completely mixed reactors." M.S. thesis, Iowa State U., 1988.
- 21. Rudolfs, W., "Effect of temeperature on sewage sludge digestion. *Industrial and Engineering Chemistry*", 19, 241, 1927.
- 22. Heukelekian, H., "Digestion of solids between thermophillic and nonthermophillic range." *Sewage Works Journal*, 3, 7, 1931.
- Heukelekian, H. and Heinemann, B., "Studies on the methane producing bacteria, I. Development of a method of enumeration." *Sewage Works Journal*, 11, 426, 1939.
- 24. Heukelekian, H. and Heinemann, B., "Studies on the methane producing bacteria, II. Enumeration in digesting sewage solids." *Sewage Works Journal*, 11, 436, 1939.
- 25. Heukelekian, H. and Heinemann, B., "Studies on the methane producing bacteria, III. Digestion of sewage solids by addition of enriched cultures of methane producing organisms." *Sewage Works Journal*, 11, 571, 1939.

- 26. Heukelekian, H. and Heinemann, B., "Studies on the methane producing bacteria, IV. Growth of methane producing bacteria in supernatant sludge liquors." *Sewage Works Journal*, 11, 965, 1939.
- 27. Heukelekian, H. and Kaplovsky, A. J., "Effect of change of temperature on thermophillic digestion." *Sewage Works Journal*, 20, 806, 1948.
- 28. McCarty, P. L., "Anaerobic waste treatment fundamentals Part II. Environmental requirements and control." *Public Works*, October 1964.
- /29. lannotti, E. L., and Fischer, J. R., "Effects of ammonia, volatile acids, pH, and sodium on the growth of bacteria isolated from a swine manure digester." *Developments in Industrial Microbiology*. Volume 25, C. S. Nash and L. A. Underkofler (eds.). Society for Industrial Microbiology, Arlington, Virginia, 1984
  - Clark, R. H., and Speece, R. E., "The pH tolerance of anaerobic digestion." *Advances in Water Pollution Research*. Volume 1. S. H. Jenkins (ed.). Pergamon Press, Oxford, 1971.
- 31. McCarty, P. L., "Anaerobic waste treatment fundamentals, part III. Toxic materials and their control." Public Works, November, 1964.
- 32. McCarty, P. L., "Anaerobic waste treatment fundamentals, part IV. Process design." *Public Works*, December, 1964.
  - 33. Speece, R. E. and McCarty, P. L., "Nutrient requirements and biological solids accumulation in anaerobic digestion." *Proc. of the First International Conference on Water Pollution Research*, London, 1962.
  - 34. Pffefer, J. T., Leiter, M., and Worlund, J. R., "Population dynamics in anaerobic digestion." *J. Water Pollution Control Federation*, 39, 1305, 1967.
  - 35. Fullen, W. J., "Anaerobic digestion of packing plant wastes." *Sewage and Industrial Wastes*, 25, 576, 1953.
  - Schroepfer, G. J., Fullen, W. J., Johnson, A. S., Ziemke, N. R., and Anderson, J. J., "The anaerobic contact process as appled to packinghouse wastes." Sewage and Industrial Wastes, 27, 460, 1955.
  - 37. Coulter, J. B., Sonedam, S., and Ettinger, M. B., "Anaerobic contact process for sewage disposal." *Sewage and Industrial Wastes*, 29, 468, 1957.
  - Steffen, A. J., "Treatment of packinghouse wastes by anaerobic digestion." Biological Treatment of Sewage and Industrial wastes, Vol. II, Reinhold Publishing Corp., New York, New York, 1958.

- 39. Taiganides, E. P., "Characteristics and treatment of wastes from a confinement hog production unit." Ph.D thesis, Iowa State U., 1963.
- 40. Hazen, T. H. and Mangold, D. W., "Functional and basic requirements of swine housing." *Agricultural Enginnering*, 41:585-590, 1960.
- 41. Taiganides, E. P. and Hazen, T. E., "Properties of farm animal excreta." *Transactions, American Society of Agricultural Engineers*, 3: 78 -80.
- 42. Dague, R. R., "Methane gas production from swine manure." *Proceedings of the Waste Management Workshop*, 115 132, American Pork Congress, Des Moines, Iowa, 1974.
- 43. Irgens, R. L. and Day, D. C., "Laboratory studies on aerobic stabilization of swine wastes." *Farm Structures Engineering Report*, University Of Illinois, January 1965.
- 44. Morrison, F.B., *Feeds & feeding*, 21st ed., Morrison Publishing Co., Ithaca, New York, 1954.
- 45. Donham, K. J., Yeggy, J., and Dague, R. R., "Chemical and physical parameters of liquid manure from swine confinement buildings: Health implications for workers, swine and the environment." *Agricultural Wastes*, 14: 97 113, 1985.
- 46. Hobson, P. N. and Shaw, B. G., "The role of strict anaerobes in the digestion of organic material." *Microbial aspects of pollution*, Sykes, G. and Skinner, F. A. (eds), Academic Press, 1971.
- 47. Hobson, P. N. and Shaw, B. G., "The anaerobic digestion of waste from an intensive pig unit." Water Research, 7: 437 - 449, 1973.
- 48. Hobson, P. N. and Shaw, B. G., "The bacterial population of piggery-waste anaerobic digesters." Water Research, 8: 507 - 516, 1974.
  - 49. Brumm, T. J. and Nye, J. C., "Dilute swine waste treatment in an anaerobic filter." *Proceedings of the 36 th Industrial waste conference, Prudue U.*, 1982.
  - 50. Ng, W. J. and Chin, K. K., "Treatment of piggery waste water by expanded bed anaerobic filters." *Biological Wastes*, 26(3): 215 232, 1988.
  - 51. Ng, W. J. and Chin, K. K., "Anaerobic filter for pig waste water." *Biological Wastes*, 20(3):157 166, 1987.
  - Colleran, E., Barry, M., Wilkie, A., and Newell, P. J., "Anaerobic digestion wastes using the upflow filter design." *Process Biochemistry*, 17(2): 12 - 17 & 41, 1982.

- 53. Wilkie, A. and Colleran, E., "Pilot scale digestion of pig slurry supernatant using an upflow anaerobic filter." *Enviromental Technology Letters*, 7: 65 - 76, 1986.
- 54. Fischer, J. R., Iannotti, E. L., and Porter, J. H. "Anaerobic digestion of swine manure at various influent solids concentrations." *Agricultural Wastes*, 11: 157 -166, 1984.
- 55. Summers, R. and Bousfield, S. "A detailed study of piggery waste anaerobic digestion." *Agricultural Wastes*, 2: 61 78, 1980.
- 56. Van Velsen, A. F. M., "Anaerobic digestion of piggery waste. 1. The influence of detention time and manure at various influent solids concentrations." *Netherlands J. of Agricultural Science*, 25: 151 - 169, 1977.
  - 57. Van Velsen, A. F. M., Lettinga, G. and den Ottelander, D., "Anaerobic digestion of piggery waste. 3. Influence of temperature." *Netherlands J. of Agricultural Science*, 27: 255 267, 1979.
  - 58. Zeeman, G., Sutter, K., Vens, T., Koster, M. and Wellinger, A., "Psycrophillic digestion of dairy cattle and pig manure: Start-up procedures for batch, fed-batch and CSTR type digesters." *Biological Wastes*, 26: 15 31, 1988.
  - 59. Cullimore, D. R., Maule, A and Mansuy, N., "Ambient temperature methanogenesis from pig waste lagoons: Thermal gradient incubator studies." *Agricultural Wastes*, 12: 147 - 157, 1985.
  - 60. Wiegant, W. M. and Zeeman, G., "The mechanism of ammonia inhibition in the thermophillic digestion of livestock wastes." *Agricultural Wastes*, 16: 243 253, 1986.
  - 61. "Standard methods for examination of water and wastewater." American Public Health Association, 16th edition, 1985.
  - 62. "Methods for chemical analysis of water and waste water." U. S. E. P. A. publication number EPA- 600/ 4 79 020, 1979.
  - 63. Dague, R. R., "A modified approach to activated sludge design." *Proceedings* of the 56th Annual Conference of the Water Pollution Control Federation, Atlanta Georgia, October 2-6, 1983.

#### ACKNOWLEDGEMENTS

The author extends his sincere appreciation to his major professor, Dr. Richard R. Dague, for his valuable guidance, suggestions and encouragement. The author would also like to thank all his colleagues for their invaluable assistance in operating the units.

The author would like to thank Dr. Dean Zimmerman and Mr. Harlan Moody for their assistance in obtaining the wastes from the Iowa State University Swine Nutrition Farm.

Finally, the author would like to thank the Department of Civil and Construction Engineering, the Biotechnology Byproducts Consortium and the U.S. Department of Agricuture for providing the financial support throughout this study and his graduate work.

88

	Total B	iogas. L/d (	@ STP_		pН	
DATE	#1	#2	#3	#1	#2	#3
9/6/90	10 1/	3 37	11.08	6 92	6 60	6 88
9/0/90 0 <i>/</i> 7/00	12.14	5.57	11.00	0.52	0.00	0.00
9/7/90	13.45	4.44 2.10	11.40	6 96	6 70	6 82
9/0/90	14.37	3.19	11.00	0.00	0.70	0.02
9/9/90	12.00	2.40	10.55	6.00	6 70	6 94
9/10/90	13.00	2.49	12.00	0.90	0.79	0.04
9/11/90	14.01	2.58	12.12	0.01	0.50	<b>C</b> 00
9/12/90	14.76	2.49	12.00	0.81	6.58	6.80
9/13/90	14.67	2.58	12.63		• • •	
9/14/90	13.84	3.46	12.07	6.64	6.42	6.65
9/15/90	14.17	3.63	11.87			_
9/16/90	13.63	3.39	9.53	6.86	6.53	6.86
9/17/90	15.21	3.91	11.65			
9/18/90	7.09	3.19	11.97	6.86	6.57	6.81
9/19/90	0.36	3.20	11.64			
9/20/90	8.28	2.76	12.11	7.06	6.64	6.82
9/21/90	12.65	2.49	12.65			
9/22/90	13.13	2.50	11.26	6.96	6.51	6.86
9/23/90	14.61	2.67	12.03			
9/24/90	15.13	2.39	12.21	6.81	6.56	6.91
9/25/90	25.28	1.41	21.04			
9/27/90	13.12	2.13	11.79	6.88	6.61	6.93
9/28/90	14.79	4.90	12.57	6.91	6.56	7.01
9/29/90	16.43	5.80	13.57	6.90	6.69	6.90
9/30/90	17.81	1.07	13.98	6.91	6.66	6.94
10/1/90	16.22	4.96	14.36	6.81	6.68	7.01
10/2/90	17.06	5.01	15.83	6.90	6.67	7.01
10/3/90	15.90	4.83	15.20	6.90	6.67	7.02
10/4/90	16.24	4.15	14.65	6.90	6.67	7.00

A. Phase - I

Total BioGas, L/d @ STP DH							
DATE	#1	#2	#3	#1	#2	#3	
10/5/90	15.25	3.53	14.63				
10/6/90	14.19	2.66	13.30	6.96	6.76	7.08	
10/7/90	14.02	3.75	12.42				
10/8/90	13.32	2.50	12.07	6.99	6.74	7.10	
10/9/90	13.82	3.48	10.79				
10/10/90	14.45	3.72	11.35	6.95	6.74	7.08	
10/11/90	13.58	1.15	11.27				
10/12/90	13.90	2.92	11.69	6.95	6.74	7.04	
10/13/90	14.59	3.34	11.78				
10/14/90	14.58	3.36	11.57	6.95	6.68	7.03	
10/15/90	15.60	3.19	11.79				
10/16/90	16.31	3.68	11.05	6.98	6.74	7.00	
10/17/90	16.01	3.78	10.38				
10/18/90	16.24	3.55	11.62	6.94	6.74	7.08	
10/19/90	16.93	3.97	12.52				
10/20/90	15.64	3.98	12.90	6.97	6.74	7.00	
10/21/90	15.90	4.00	11.37				
10/22/90	17.46	4.34	14.53	7.04	6.76	7.05	

B. Phase - II

.

	Total I	Biogas, L/d	@ STP		Hq	
DATE	#1	#2	#3	#1	#2	_#3
10/26/90	0.00	0.00	0.00	7.01	6.70	7.10
10/27/90	11.50	3.50	8.90	7.01	6.70	7.10
10/28/90	17.40	4.80	14.40			
10/29/90	11.70	2.80	6.10	7.00	6.69	7.02
10/30/90	23.40	4.50	9.90	7.01	6.70	7.07
10/31/90	25.60	4.70	12.00	7.04	6.67	7.06
11/1/90	28.50	7.80	11.20	7.07	6.77	7.09

	Tota	l Biogas, L/	d@STP		pН	
DATE	#1	#2	#3	#1	#2	#3
11/2/90	26.90	8.00	22.00	7.09	6.80	7.14
11/3/90	27.70	9.50	22.40	7.09	6.86	7.13
11/4/90	29.10	10.70	24.40	7.09	6.86	7.13
11/5/90	34.30	10.40	25.00	7.14	6.91	7.20
11/6/90	36.50	11.00	26.60	7.16	6.93	7.13
11/7/90	34.08	10.48	23.79	7.17	6.89	7.18
11/8/90	36.45	11.73	23.36	7.21	6.94	7.21
11/9/90	33.85	9.63	23.58	7.18	6.90	7.18
11/10/90	28.33	8.16	19.81	7.11	6.86	7.13
11/11/90	25.74	8.07	19 <b>.01</b>	7.11	6.84	7.21
11/12/90	23.01	7.49	17.60	7.15	6.83	7.10
11/13/90	22.00	7.48	15.96	6.99	6.80	7.08
11/14/90	22.44	6.97	15.65	7.06	6.83	7.04
11/15/90	21.95	7.23	6.96	7.11	6.80	7.05
11/16/90	21.74	8.35	12.75	7.09	6.78	7.05
11/17/90	20.97	8.31	14.10	7.08	6.77	7.08
11/18/90	21.80	6.73	12.92	7.08	6.77	7.11
11/19/90	22.14	7.85	12.14	7.10	6.73	7.05
11/20/90	21.69	8.53	12.36	6.96	6.71	6.95
11/21/90	20.00	7.88	14.07	7.05	6.73	6.98
11/22/90	19.16	7.18	11.97	7.00	6.72	7.01
11/23/90	20.60	5.59	13.05	7.01	6.74	6.99
11/24/90	20.96	4.69	13.88	7.00	6.71	7.00
11/25/90	21.40	7.93	13.21	7.00	6.79	7.00
11/26/90	19.31	7.28	15.18	7.04	6.85	6.96
11/27/90	21.26	8.93	15.72	7.04	6.84	7.00
11/28/90	22.54	8.42	17.20	7.05	6.86	7.06
11/29/90	23.51	9.51	18.22	7.05	6.84	7.05
11/30/90	23.30	9.37	17.94	7.15	6.83	7.08
12/1/90	22.57	9.35	19.60	7.16	6.83	7.06

	Tota	l Biogas, L/	d@STP		pН	
DATE	#1	#2_	#3	#1	#2	#3
12/2/90	25.03	10.17	20.35	7.17	6.83	7.00
12/3/90	24.98	10.14	19.82	7.14	6.83	7.08
12/4/90	26.23	9.28	19.83	7.04	6.78	7.08
12/5/90	26.49	9.97	19.49	7.03	6.79	7.03
12/6/90	34.89	10.58	24.93	7.03	6.76	7.00
12/7/90	29.71	9.99	19.27	6.99	6.81	7.05
12/8/90	26.54	9.92	16.62	7.05	6.76	7.02
12/9/90	23.23	9.11	16.26	7.01	6.78	7.01
12/10/90	22.47	9.06	15.72	6.96	6.75	7.02
12/11/90	23.81	8.50	14.87	7.06	6.70	7.02
12/12/90	23.51	8.13	13.94	7.00	6.76	7.07
12/13/90	21.83	8.41	13.69	7.01	6.70	7.03
12/14/90	22.22	5.78	13.69	7.01	6.72	7.04
12/15/90	23.87	11.22	12.92	7.01	6.70	6.99
12/16/90	21.92	8.70	10.65	7.04	6.70	6.96
12/17/90	21.47	8.10	14.43	7.01	6.72	7.00
12/18/90	22.28	8.04	16.44	7.01	6.72	7.03
12/19/90	21.19	6.94	16.02	6.94	6.74	6.97
12/20/90	20.69	6.87	14.98	7.08	6.72	7.03
12/21/90	20.69	7.43	14.87	7.05	6.70	7.02
12/22/90	20.45	7.48	15.76	7.05	6.70	7.03
12/23/90	25.86	10.54	18.29	7.04	6.71	7.01
12/24/90	23.63	9.56	19.03	7.05	6.73	7.01
12/25/90	27.18	10.18	21.27	7.08	6.74	7.03
12/26/90	24.46	9.06	17.48	6.98	6.74	7.02
12/27/90	30.66	10.87	19.70	7.01	6.70	6.99
12/28/90	32.28	10.94	20.72	7.04	6.72	7.02
12/29/90	31.33	10.80	22.15	7.05	6.73	7.03
12/30/90	29.75	11.39	19.80			
12/31/90	25.46	11.60	19.16	7.10	6.79	7.05

Phase II (continued)

	Tota	al Biogas. L/	d@STP		H	
DATE	#1	#2	#3	#1	#2	#3
1/1/91	29.21	11.58	19.90	7.12	6.80	7.02
1/2/91	30.00	10.49	19.70			
1/3/91	30.09	10.91	20.27			
1/4/91	30.29	10.91	20.01	7.13	6.79	7.05
1/5/91	29.64	11.03	21.24			
1/6/91	31.77	11.78	22.19			
1/7/91	32.96	12.41	22.28			
1/8/91	29.51	12.09	21.75	7.10	6.87	7.16
1/9/91	30.29	11.39	19.17			
1/10/91	30.89	11.19	20.32	7.21	6.70	7.09
1/11/91	30.55	11.34	20.36			
1/12/91	30.58	11.11	20.27			
1/13/91	29.72	10.70	19.55			

Phase II.(continued)

C. Phase - III

	<u>lotal</u>	Biogas, L/C			pH	
DATE	#1	#2	#3	#1	#2	<u>#3</u>
1/17/91	1.96	1.07	3.21			
1/18/91	12.79	3.04	7.87			
1/19/91	14.30	0.98	11.10			
1/20/91	15.18	4.53	11.36	6.97	6.74	6.92
1/21/91	16.38	4.50	10.35			
1/22/91	17.24	4.82	9.47			
1/23/91	17.35	4.69	9.74	6.77	6.49	6.70
1/24/91	17.56	4.39	10.48	6.84	6.52	6.78
1/25/91	16.52	5.15	11.38	6.84	6.69	6.84
1/26/91	16.55	4.95	10.43	6.80	6.53	6.77
1/27/91	17.59	4.97	12.97	6.78	6.57	6.82
1/28/91	18.59	5.05	12.22	6.86	6.62	6.80
1/29/91	18.49	4.47	11.88	6.88	6.66	6.83

					-	
-	Tota	Biogas. L/	d@STP		pН	
DATE	#1	#2	#3	#1	#2	#3
1/30/91	17.10	5.10	12.35			
1/31/91	16.40	4.66	11.83			
2/1/91	16.71	5.12	12.40	7.03	6.80	6.92
2/2/91	16.36	4.85	12.14			
2/3/91	17.15	5.03	12.30	6.94	6.77	6.92
2/4/91	17.62	4.92	12.97			
2/5/91	18.21	5.20	12.56	7.13	6.99	7.11
2/6/91	17.89	4.86	12.32			
2/7/91	16.78	4.87	12.54	7.02	6.80	6.94
2/8/91	17.26	4.94	12.22			
2/9/91	17.16	5.09	12.69	7.04	6.88	6.98
2/10/91	16.50	4.75	12.37			
2/11/91	19.68	6.29	14.38			
2/12/91	20.46	5.90	15.10			
2/13/91	20.83	7.06	15.44	7.04	6.82	_6.95
		۵	). Phase - IV			
	Total	Biogas, L/	d@STP		рН	
DATE	#1	#2	#3	#1	#2	#3
2/14/91	21.61	6.46	14.08			
2/15/91	22.42	6.66	15.76			
2/16/91	18.42	5.45	13.59	7.04	6.80	6.97
2/17/91	18.55	5.48	13.43			
2/18/91	17.50	5.48	13.34			
2/19/91	16.62	5.42	11.73	6.93	6.68	6.79
2/20/91	16.22	4.99	12.30			
2/21/91	12.90	8.18	14.41	6.81	6.58	6.77
2/22/91	14.56	8.81	15.01			
2/23/91	9.69	9.78	16.31			

Phase III.(continued)

<u></u>	 Tota	I Biogas, L/c	d@STP		DΗ	
DATE	#1	#2	#3	#1	#2	#3
2/24/91	14.92	9.95	17.36	6.85	6.77	6.87
2/25/91	13.62	12.08	19.07			
2/26/91	15.46	13.11	22.15			
2/27/91	14.62	13.28	21.58	6.95	6.91	6.96
2/28/91	15.48	13.89	20.96			
3/1/91	14.70	14.18	19.87			
3/2/91	16.81	15.14	22.97	6.95	6.93	7.02
3/3/91	16.43	12.84	20.66			
3/4/91	19.76	13.94	24.67			
3/5/91	17.66	13.90	23.35	6.91	6.85	6.95
3/6/91	17.64	14.66	23.78			
3/7/91	18.49	14.76	24.45			
3/8/91	18.47	14.74	19.89	6.98	6.87	6.96
3/9/91	17.93	13.56	22.13			
3/10/91	17.76	14.35	23.05			
3/11/91	17.98	15.22	22.87	6.93	6.87	6.97
3/12/91	17.16	15.05	17.69			
3/13/91	16.64	16.82	22.19			
3/16/91	19.31	16.61	22.99	6.80	6.75	6.84
3/17/91	19.02	17.14	21.52			
3/18/91	17.87	15.11	20.98			
3/19/91	20.58	15.86	20.76			
3/20/91	21.91	16.43	21.29			
3/21/91	22.36	16.92	22.09	6.83	6.83	6.88
3/22/91	22.70	17.18	23.93			
3/23/91	21.10	17.78	23.21	6.77	6.72	6.82
3/24/91	21.23	17.34	21.85			
3/25/91	22.41	18.05	23.47	6.79	6.77	6.85
3/26/91	22.70	17.88	23.92			
3/27/91	19.48	16.89	22.25	6.76	6.68	6.78

Phase IV. (continued)

	Total	Biogas, L/c	@ STP	pH			
DATE	#1	#2	#3	#1	#2	#3	
3/28/91	19.47	16.16	21.31				
3/29/91	19.79	15.97	22.01	7.00	6.83	6.94	
3/30/91	18.98	16.29	22.83				
3/31/91	18.91	15.77	22.76	6.83	6.74	6.84	
4/1/91	19.20	16.24	26.37				
4/2/91	20.37	15.70	25.93				
4/3/91	21.13		24.62	6.87	7.08	6.94	
4/4/91	20.48	16.01	24.33				
4/5/91	20.62	16.53	24.00	6.94	6.90	6.98	
4/6/91	24.33	18.54	24.69				
4/7/91	24.63	18.45	24.19				
4/8/91	23.55	18.68	13.38	6.95	6.90	7.00	
4/9/91	15.70	12.26	14.64				
4/10/91	13.96	10.71	14.32				
4/11/91	5.25	9.23	11.67	6.87	6.76	6.86	
4/12/91	19.86	8.76	11.38				
4/17/91	20.04	20.22	27.72	6.91	6.85	6.94	
4/18/91	10.60	8.62	10.78				
4/19/91	21.42	17.84	18.91	6.95	6.92	6.97	
4/20/91	21.63	16.04	18.47				
4/21/91	23.74	15.88	20.94	6.98	6.91	6.95	
4/22/91	27.05	19.43	21.67				
4/23/91	26.91	18.54	22.90				
4/24/91	25.89	20.43	22.84	7.01	6.94	6.98	

Phase IV. (continued)

## APPENDIX B. CHEMICAL PARAMETERS DATA

## A. Phase - I

## Reactor #1

DATE	Total Alkalinity <sup>a</sup>	Volatile Acids <sup>b</sup>	Ammonia <sup>c</sup>
9/4/90	3942	40	150
9/11/90	3870	150	240
9/18/90	3250	130	260
9/25/90	3490	80	350
10/2/90	3120	50	330
10/9/90	2710	30	370
10/19/90	2580	25	400

<sup>a</sup> Total alkalinity as mg/L Ca CO<sub>3</sub> <sup>b</sup> Total volatile acids as mg/L CH<sub>3</sub>COOH <sup>c</sup> Ammonia as mg/L N

DATE	Total Alkalinity	Volatile Acids	Ammonia
9/4/90	620	50	140
9/11/90	840	32	240
9/18/90	1150	28	340
9/25/90	1040	22	310
10/2/90	1080	12	310
10/9/90	1120	8	305
10/19/90	1080	8	310

Reactor	#3
---------	----

DATE	Total Alkalinity	Volatile Acids	Ammonia
9/4/90	3790	110	110
9/11/90	3400	195	160
9/18/90	3280	27	315
9/25/90	2860	65	342
10/2/90	2910	27	355
10/9/90	2750	25	428
10/19/90	2680	24	400

### B. Phase - II

## Reactor #1

DATE	Total Alkalinity	Volatile Acids	Ammonia
11/2/90	2780	48	420
11/9/90	2640	54	430
11/16/90	2690	40	470
11/30/90	2740	35	540
12/7/90	2840	37	580
12/15/90	2810	40	640
12/22/90	2940	32	720
12/29/90	2910	35	860
1/5/91	2885	31	820
1/15/91	2900	31	825

.

DATE	Total Alkalinity	Volatile Acids	Ammonia
11/2/90	1180	9	210
11/9/90	1095	11	265
11/30/90	1350	10	259
12/7/90	1350	13	281
12/15/90	1420	16	250
12/22/90	1440	15	310
12/29/90	1510	16	315
1/5/91	1470	16	295
1/15/91	1455	16	291

DATE	Total	Volatile	Ammonia
	Alkalinity	Acids	
<u> </u>			
11/2/90	2450	28	410
11/9/90	2560	25	422
11/16/90	2480	26	510
11/30/90	2470	31	530
12/7/90	2400	26	515
12/15/90	2450	25	560
12/22/90	2440	26	589
12/29/90	3240	28	567
1/5/91	3390	29	575
1/15/91	3240	28	591

# C. Phase - III

# Reactor #1

DATE	Total Alkalinity	Volatile Acids	Ammonia
1/21/91	3175	36	780
1/28/91	3850	75	560
2/4/91	4230	79	490
2/11/91	4350	75	463

# Reactor #2

DATE	Total Alkalinity	Volatile Acids	Ammonia
1/21/91	1750	28	285
1/28/91	2490	26	240
2/4/91	2540	36	160
	2710	39	138

DATE	Total Alkalinity	Volatile Acids	Ammonia
1/21/91	3240	28	385
1/28/91	3390	28	425
2/4/91	3460	35	490
_2/11/91	3380	41	588

## D. Phase - IV

DATE	Total Alkalinity	Volatile Acids	Ammonia
2/18/91	4080	79	475
2/25/91	3860	62	460
3/4/91	4150	65	538
3/11/91	4350	86	567
3/19/91	3640	110	549
3/26/91	3350	142	630
4/3/91	3240	162	615
4/20/91	3260	160	595

### Reactor #1

DATE	Total Alkalinity	Volatile Acids	Ammonia
2/18/91	1600	21	165
2/25/91	1760	45	179
3/4/91	2320	39	189
3/11/91	2100	45	165
3/19/91	2330	59	134
3/26/91	2330	62	175
4/3/91	2400	69	148
4/20/91	2500	85	321

DATE	Total Alkalinity	Volatile Acids	Ammonia		
2/18/91	3260	40	315		
2/25/91	3200	55	369		
3/4/91	3175	69	435		
3/11/91	3070	52	495		
3/19/91	2780	64	512		
3/26/91	2890	75	361		
4/3/91	2900	40	455		
4/20/91	2810	40	446		

Reactor #3

٠

.

### 103

# APPENDIX C. SOLIDS AND SRT DATA

## A. Phase- I

### Reactor #1

Date	MLSS <sup>a</sup>	MLVSS	Inf. TSS	Inf. VSS	Eff. TSS	Eff. VSS	SRT
9/11/90	14230	10150	18320	17550	7320	6060	10.05
9/17/90	12550	9260	18970	16480	6560	5820	9.55
9/21/90	13050	9600			6510	4820	11.95
9/24/90	12510	9070			6970	5050	10.78
9/25/90	12850	9360	21940	18450	6600	5600	10.03
10/5/90	11240	9440			6650	4260	13.30
10/7/90	11800	9340	19330	17450	6090	4020	13.94
10/9/90	10570	9570			5000	4250	13.51
10/11/90	10580	9190			6580	5280	10.44
10/13/90	10580	9520	· · · · · · · · · · · · · · · · · · ·		6020	4250	13.44
~							

<sup>a</sup> All values are in mg/L

Date	MLSS	MLVSS	Inf. TSS	Inf. VSS	Eff. TSS	Eff. VSS	SRT
9/21/90	5640	3260			490	310	69.06
9/24/90	6120	4560			750	610	48.96
9/25/90	7100	4600	7310	6150	580	540	73.45
10/5/90	6980	4840			410	360	102.15
10/7/90	7430	5430			350	320	127.37
10/9/90	7030	5260	6440	5600	410	370	102.88
10/11/90	7620	5730			450	360	101.60
10/13/90	7890	5900			460	440	<u>102.91</u>

Reactor a	#3
-----------	----

Date	MLSS	MLVSS	Inf. TSS	Inf. VSS	Eff. TSS	Eff. VSS	SRT
9/11/90	12460	8950			5590	4930	10.89
9/17/90	12550	9160	20370	16485	5620	4950	11.10
9/21/90	12350	9200			6680	4930	11.20
9/24/90	13100	9580			6970	5050	11.38
9/25/90	13310	9700	21940	18450	7680	5630	10.34
10/5/90	14040	9400			7700	5980	9.43
10/7/90	13800	9600	19330	17450	8250	5850	9.85
10/9/90	11870	8720			4040	2670	19.60
10/11/90	12550	8740			4440	3330	15.75
<u>10/13/90</u>	12820	9510			4580	3280	17.40

B. Phase- II

.

	Date	MLSS	MLVSS	Inf. TSS	Inf. VSS	Eff. TSS	Eff. VSS	SRT
•	11/6/90	12910	11424			7890	6410	10.69
1	1/18/90	12120	11580			7640	6210	11.19
1	2/11/90	12400	9480	21860	18630	6910	5330	10.67
1:	2/24/90	16400	12000			7850	6490	11.09
	1/6/91	18130	14440	25470	23810	8540	6035	14.36
	1/8/91	18695	15070	24550	22330	8830	7080	12.77
	1/10/91_	18240	14700	24570	22360	8760	6720	13.13
Reactor	#2							
---------	----							
---------	----							

Date	MLSS	MLVSS	Inf. TSS	Inf. VSS	Eff. TSS	Eff. VSS	SRT
11/6/90	7900	6340			2140	1980	19
11/18/90	9560	7820			2230	1410	33
12/11/90	10530	8050	8740	7452	1570	1220	18
12/24/90	11110	8550			3570	2710	20
1/6/91	11140	9010	10190	9524	3400	2610	20
1/8/91	11410	9410	9820	8932	3350	2690	21
1/10/91	10830	8950	9828	8944	3270_	2800	19

Reactor #3

Date	MLSS	MLVSS	Inf. TSS	Inf. VSS	Eff. TSS	Eff. VSS	SRT
11/6/90	14650	12620			3810	2950	26
11/18/90	15430	12240			3650	2820	26
12/11/90	18320	13460	17488	1490	4 3540	2600	31
12/24/90	18980	15720			7340	6150	15
1/6/91	17890	13980	20376	5 1904	8 8120	6400	13
1/8/91	18915	14880	19640	1786	4 7600	5970	14
1/10/91_	14520	11680	19656	1788	8 7550_	5970	12

C. Phase - III

Date	MLSS	MLVSS	Inf. TSS	Inf. VSS	Eff. TSS	Eff. VSS	SRT
1/22/91	20220	15730			6790	5030	19
1/24/91	19940	16240			6170	4850	20
1/29/91	19070	15280	14310	12980	5720	4380	21
2/4/91	20590	15770	13880	13030	5770	4700	20
2/6/91	20300	16440	14270	13320	6020	4420	22
2/8/91	20200	15420	12980	11900	5580	4600	20

Reactor #2

Date	MLSS	MLVSS	Inf. TSS	Inf. VSS	Eff. TSS	Eff. VSS	SRT
1/22/91	10970	9210			1600	1450	38
1/24/91	11050	9210			930	880	63
1/29/91	10950	9260	4770	4327	850	750	74
2/4/91	10070	8120	4630	4340	570	440	111
2/6/91	10190	8640	4755	4440	530	480	108
<u>2/8/91</u>	10560	8780	4330	3970	570	495	106

Reactor #3

Date	MLSS	MLVSS	Inf. TSS	Inf. VSS	Eff. TSS	Eff. VSS	SRT
1/22/91	14140	11500			3390	3120	22
1/24/91	18120	14160			2090	2680	32
1/29/91	18310	14400	9540	8653	2740	2090	41
2/4/91	17990	14380	9254	8680	2970	2050	42
2/6/91	17440	14450	9510	8883	2590	2190	40
2/8/91	15610	14210	8650	7935	2510	2100	41

### D. Phase - IV

Reactor #1

Date	MLSS	MLVSS	Inf. TSS	Inf. VSS	Eff. TSS	Eff. VSS	SRT
3/17/91	22390	16430			6790	5590	18
3/19/91	22410	19710	28850	27850	6160	4950	24
3/22/91	23710	18790			6720	5680	20
3/28/91	22280	16550			7660	6130	16
4/1/91	24250	18560	29330	28230	7290	6210	18
4/7/91	20410	15590			9300	7660	12
4/21/91	31630	24880	32160	29842	9450	7470	20
<u>4/23/91</u> _	27720	25310	29860	27820	9610	7760	20

Reactor	#2
reactor	π <u></u>

Date	MLSS	MLVSS	Inf. TSS	Inf. VSS	Eff. TSS	Eff. VSS	SRT
2/28/91	12490	10830			6860	5800	11
3/4/91	12420	10360			6480	5400	11
3/11/91	13290	11480	20278	19228	5610	4680	15
3/17/91	13510	11700			5510	4650	15
3/19/91	14550	12670	20262	19397	5140	4460	17
3/22/91	15320	13240			4570	3660	22
3/28/91	13130	10510			4590	3910	16
4/1/91	14840	12390	22886	22155	4390	3510	21
4/7/91	12600	10460			5880	4950	13
4/21/91	21580	17630	21318	19779	8530	6770	16
<u>4/23/91</u>	19120	15700	19423	18226	7880	6180	15

Reactor #3

Date	MLSS	MLVSS	Inf. TSS	Inf. VSS	Eff. TSS	Eff. VSS	SRT
2/28/91	16850	13490			6070	5170	16
3/4/91	22930	18110			8260	6850	16
<b>3/11/</b> 91	23340	18380	27040	25640	7940	6670	16
3/17/91	20440	15940			7820	6510	15
3/19/91	20820	16040	27110	25990	7110	5960	16
3/22/91	26280	20290			7460	6210	20
3/28/91	25490	19740			5990	4990	24
4/1/91	25300	18970	27240	26270	8770	7000	16
4/7/91	16010	12690			6980	5820	13
4/21/91	23570	19650	28420	26370	9030	8380	14
4/23/91	24530	<u>19740</u>	26090	24300	8960	7310	16

# APPENDIX D. COD DATA

#### A. Phase - I

#### Reactor #1

Date	Influent.	Influent.	Mixed Liquo	r Effluent	Effluent
	Total CODª	Soluble CC	D Total COD	Total COD	
9/3/90	17880	4660	15060	11630	1577
9/13/90	19410	7020	11510	11390	1710
9/22/90	18670	6490	11690	8900	1460
9/30/90	18460	6900	12840	6100	1120
10/9/90	19500	6300	12820	8140	900
10/10/90	19700	6070	12870	6510	900
10/11/90	19760	6760	12840	6510	850
10/12/90	19230	6030	12840	6540	1020
10/22/90	19450	6270	12860	6420	830

a All values are in mg/L

.

Date	Influent.	Influent.	Mixed Liquor	Effluent	Effluent
	Total COD	Soluble COD	Total COD	Total COD	Soluble COD
9/3/90	5960	1550	5970	4410	620
9/13/90	6470	2340	6010	3250	450
9/22/90	6220	2160	6050	2710	490
9/30/90	6750	2490	6000	1550	400
10/9/90	6170	2100	6070	1420	410
10/10/90	6270	2020	6050	1250	410
10/11/90	6040	2250	6030	1320	420
10/12/90	5700	2010	6080	1340	445
10/22/90	6150	3090	6080	1570	420

Re	actor	#3
----	-------	----

Date	Influent.	Influent.	Mixed Liquo	r Effluent	Effluent
	Total COD	Soluble CO	D Total COD	Total COD	Soluble COD
9/3/90	17880	4660	12860	17410	2060
9/13/90	19410	7020	11520	9440	1970
9/22/90	18670	6490	11640	8660	1840
9/30/90	18460	6900	11780	7020	2130
10/9/90	19500	6300	11820	6760	1140
10/10/90	19700	6070	11790	6540	770
10/11/90	19760	6760	11830	6980	1100
10/12/90	19230	6030	11800	7020	1200
10/22/90	19450	6270	11810	6380	1420

#### B. Phase - II

Date	Influent.	Influent.	Mixed Liquor	Effluent	Effluent
	Total COD	Soluble CO	DD Total COD	Total COD	Soluble COD
11/12/90	23960	11020	12020	10260	1410
11/27/90	24640	11500	12400	9840	1470
12/11/90	26750	12470	12760	8720	1340
12/29/90	32890	10500	13100	10690	1240
1/3/91	32680	10560	14600	10550	1250
1/7/91	32610	10150	15490	10150	1240
1/9/91	32410	10570	15890	10370	1220
1/11/91	32480	8920	16200	10230	1310

Date	Influent.	Influent.	Mixed Liquor	Effluent	Effluent
	Total COD	Soluble COE	Total COD	Total COD	Soluble COE
11/12/90	11980	5510	11440	4140	750
11/27/90	12320	5750	11560	4270	760
12/11/90	10700	4990	11390	3960	640
12/29/90	12170	4200	11490	3680	680
1/3/91	13500	3520	11510	3745	580
1/7/91	13040	3380	11490	3610	550
1/9/91	12930	3520	11510	3700	550
1/11/91	13400	3570	11470	3750	670

Reactor #2

Re	actor	#3
----	-------	----

Date	Influent.	Influent.	Mixed Liquor	Effluent	Effluent
	Total COD	Soluble COD	Total COD	Total COD	Soluble COD
11/12/90	23960	11020	12600	10260	1410
11/27/90	24640	11500	13600	9840	1470
12/11/90	21400	9980	15640	9780	1340
12/29/90	24340	8400	15480	9250	1270
1/3/91	26100	7040	15530	8960	1250
1/7/91	26410	6760	15540	9160	1210
1/9/91	26230	7040	15560	8670	1290
1/11/91	26180	7140_	15590	8890	1390

### C. Phase - III

$\pi = \pi = \pi$	R	ea	ctc	r	#1
-------------------	---	----	-----	---	----

Date	Influent.	Influent.	Mixed Liquor	Effluent	Effluent
	Total COD		Total COD	Total COD	Soluble COD
1/23/91	19050	6680	9360	8650	1120
2/4/91	19330	7040	10070	6130	910
2/6/91	21350	6630	10640	6020	980
2/8/91	20890	6840	11090	6110	840
		React	or #2		
Date	Influent.	Influent.	Mixed Liquor	Effluent	Effluent
	Total COD	Soluble COD	Total COD	Total COD	Soluble COD
1/23/91	6160	2130	10130	3200	390
2/4/91	6040	2360	9930	2390	350
2/6/91	6400	2210	9800	2330	330
<u>   2/8/91    </u>	6220	2000	8720	2520	340

Date	Influent.	Influent.	Mixed Liquor	Effluent	Effluent
	Total COD	Soluble CO	D Total COD 1	Total COD	Soluble COD
1/23/91	12150	3990	11410	3430	720
2/4/91	12080	4420	11520	2910	670
2/6/91	12800	4730	11480	3020	660
2/8/91	12440	4360	11740	3080	680

# D. Phase- IV

Reactor	#1	
---------	----	--

Date	Influent.	Influent.	Mixed Liquor	Effluent	Effluent
	Total COD	Soluble CO	D Total COD	Total COD	Soluble COD
3/10/91	27195	8950	13400	2783	
3/20/91	27720	8840	13600	2600	
4/1/91	32790	11940	23490	12150	2010
4/7/91	34200	9960	12930	2510	
4/21/91	34920	8610	20590	12860	2660
4/23/91	34100	8640	12800	2400	
		React	or #2		
Date	Influent.	Influent.	Mixed Liquor	Effluent	Effluent
	Total COD	Soluble COE	Total COD	Total COD	Soluble COD
3/10/91	19150	6710	17650	8200	820
3/21/91	19590	6430		7650	790
4/1/91	21860	7960	18490	6490	1140
4/7/91	22800	6640		8010	1080
4/21/91	23280	5700	19670	10554	775
4/23/91	22730	5720	21570	11100	1070
Reactor #3					
Date	Influent.	Influent.	Mixed Liquor	Effluent	Effluent
	Total COD	Soluble COE	) Total COD	Total COD	Soluble COD
3/10/91	29260	7560	20580	11400	1120
3/21/91	30490	10480	12880	1280	
4/1/91	30560	10650	21640	11210	1100
4/7/91	29040	8880	15230	10390	1150
<b>4/21/</b> 91	29590	7720	23610	11800	950
4/23/91	30550	7630	23630	11570	1150

### APPENDIX E. METHANE PRODUCTION DATA

	Methane Production, L/day @ STP		
Date	#1	#2	#3
9/11/90	8.81	1.69	7.21
9/18/90	4.45	2.29	7.35
9/27/90	8.24	1.53	7.20
10/3/90	9.88	3.41	9.44
10/10/90	9.01	2.97	7.09
10/17/90	9.94	3.17	8.41
10/22/90	10.81	3.11	8.95

# A. Phase- I

# B. Phase- II

# Methane Production, L/day @ STP

Date	#1	#2	#3	_
10/26/90	0.00	0.00	0.00	
10/30/90	7.30	2.00	3.73	
11/3/90	16.87	5.61	13.31	
11/6/90	21.51	7.30	15.45	
11/10/90	21.12	6.86	14.65	
11/13/90	14.27	5.38	11.10	
11/17/90	13.63	5.86	7.80	
11/21/90	13.41	6.03	7.60	
11/24/90	12.77	3.96	8.05	
11/28/90	13.14	6.40	9.64	
12/1/90	14.47	6.62	10.98	
12/5/90	16.08	6.31	12.28	
12/8/90	18.39	6.83	12.05	

	Methane Production, L/day @ STP		
Date	#1	#2	#3
12/11/90	14.00	6.51	9.62
2/14/90	13.73	5.31	8.64
12/17/90	13.72	5.57	6.52
12/20/90	13.22	4.50	9.90
12/24/90	16.14	6.86	11.47
12/27/90	15.36	5.72	10.70
12/31/90	18.65	7.36	12.16
1/3/91	18.63	6.44	12.33
1/7/91	19.86	7.34	13.71
1/14/91	18.63	6.65	12.00

Phase- II Data(continued)

C. Phase - III

	Methane	Methane Production, L/day @ STF		
Date	#1	#2	#3	
1/23/91	10.41	3.20	6.05	
1/27/91	10.69	3.35	8.10	
1/30/91	10.27	3.41	7.71	
2/3/91	10.43	3.37	7.77	
2/8/91	10.57	3.28	7,71	

.

	Methane Production, L/day @ STP			
Date	#1	#2	#3	-
2/13/91	12.50	4.62	9.59	
2/24/91	9.11	6.23	10.62	
3/8/91	11.30	8.78	12.11	
3/13/91	10.09	9.71	12.94	
3/18/91	13.15	11.05	15.50	
3/25/91	12.86	10.38	13.90	
4/1/91	11.11	9.67	15.69	
4/5/91	11.61	9.96	13.64	
4/7/91	14.54	11.03	15.12	
4/9/91	9.62	7.49	8.93	
4/18/91	7.04	5.95	7.35	
4/21/91	15.52	10.56	13.74	
4/22/91	15.94	13.41	14.61	
4/23/91	15.99	11.43	13.64	
4/24/91	15.50	12.00	13.14	_

D. Phase- IV Data