

Effects of particle size reduction on anaerobic digestion:

Comparison of conventional and two-phase systems

by

Wade Alan Weiss

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: Sanitary Engineering

Signatures have been redacted for privacy

Iowa State University
Ames, Iowa

1990

TABLE OF CONTENTS

	Page
LIST OF ABBREVIATIONS	ix
ACKNOWLEDGEMENTS	x
CHAPTER I. INTRODUCTION	1
CHAPTER II. OBJECTIVES AND SCOPE OF THE STUDY	3
CHAPTER III. BACKGROUND	5
Anaerobic Degradation	5
Process Control	9
Modes of Operation	14
High Rate Digestion	17
Phase Separation	18
Particle Size	21
CHAPTER IV. EXPERIMENTAL STUDY	25
Experimental Design	25
System Operation	31
Analytical Techniques	36
CHAPTER V. RESULTS AND DISCUSSION	49
Methane Production	49
Volatile Acids Analysis	52
Particle Size Fractionation	56
CHAPTER VI. CONCLUSIONS AND RECOMMENDATIONS	67
BIBLIOGRAPHY	69
APPENDIX A. DAILY REACTOR FEEDING PROCEDURES	73
Appendix A-1. Daily Data Recording and Sampling	75

	Page
Appendix A-2. Daily Feeding Procedure for the Conventional System (Reactor 3)	78
Appendix A-3. Morning Feeding Procedure for the Two-phase System Acidogenic Reactor (Reactor 1)	81
Appendix A-4. Daily Feeding Procedure for the Two-phase System Methanogenic Reactor (Reactor 2)	84
Appendix A-5. Afternoon and Evening Feeding Procedure for the Two-phase System Acidogenic Reactor (Reactor 1)	86
APPENDIX B. DATA COLLECTED DURING THE EXPERIMENTAL STUDY	88
Appendix B-1. Daily Operating Data during Primary Sludge Feed Study of Phase I	90
Appendix B-2. Daily Operating Data during Homogenized Feed Study of Phase I	97
Appendix B-3. Daily Operating Data during Homogenized Feed Study of Phase II	104
Appendix B-4. Daily Operating Data during Primary Sludge Feed Study of Phase II	111
Appendix B-5. Daily Operating Data during Primary Sludge Feed Study of Phase III	118
Appendix B-6. Daily Operating Data during Homogenized Feed Study of Phase III	125
APPENDIX C. DATA USED IN CALCULATION OF GAS PRODUCTION RATE PER GRAM OF COD	129
APPENDIX D. SUMMARY OF DATA COLLECTED DURING PARTICLE SIZE FRACTIONATION STUDIES	133
APPENDIX E. SUMMARY OF ANALYSIS PERFORMED FOR INDIVIDUAL VOLATILE ORGANIC ACIDS IN EACH REACTOR	138
APPENDIX F. SUMMARY OF RESULTS OF PHASE II PARTICLE SIZE FRACTIONATION STUDIES	141
APPENDIX G. DATA USED IN CALCULATION OF HYDROLYSIS RATE CONSTANTS	145

LIST OF TABLES

	Page
Table 1. Requirements for anaerobic digestion (20)	9
Table 2. Indicators of impending digester failure (20)	10
Table 3. Concentrations of inorganics reported to be toxic to anaerobic digestion	13
Table 4. Design criteria for high rate anaerobic digestion systems	18
Table 5. Summary of high rate anaerobic treatment systems operating in the approximate range of 10 to 20 days HRT	19
Table 6. Summary of reactor design parameters from Holland and Chapman (12)	26
Table 7. Operating parameters analyzed for feed and effluent from each system	32
Table 8. Values for SRT of each system and length of individual studies for each phase of the experimental study	34
Table 9. Characteristics of feed sludge	37
Table 10. Summary of analyses performed and frequency of measurement	39
Table 11. Summary of individual COD fractions	42
Table 12. Summary of values used in calculating the equivalent spherical diameter of the smallest particle removed in each step of fractionation	44
Table 13. Summary of cumulative particle size fractions	44
Table 14. Summary of incremental particle size fractions	45
Table 15. Operating conditions for gas analysis	46
Table 16. Operating conditions for individual volatile organic acid analysis	48

	Page
Table 17. Average methane production rates for anaerobic treatment systems during each phase of the experimental study	50
Table 18. Values for calculated methane production for each treatment system during the 15-day and 20-day HRT phases of the experimental study	53
Table 19. Comparison of the individual volatile organic acids concentrations in the conventional and methanogenic reactors during phase II	55
Table 20. Summary of incremental particle size fractions	57
Table 21. Percent COD removal for each treatment system during the 10-day and 15-day HRT phases of the experimental study	58
Table 22. Percent COD contained in incremental fractions of samples from each reactor during the primary sludge feed study of phase III	61
Table 23. Percent COD contained in incremental fractions of samples from each reactor during the homogenized sludge feed study of phase III	63
Table 24. Hydrolysis rate constants for the acidogenic reactor of the two-phase system during both feed studies of phases II and III	66
Table A-1. Summary of reactors feeding schedule	73
Table A-2. Summary of sampling parameters and frequency of measurement	75
Table A-3. Distance conventional reactor level drops in relation to various volumes of sludge withdrawn at specified detention times	79
Table A-4. Approximate volume of sludge to be withdrawn from reactor 1 for feed into reactor 2 at specified detention time	82
Table A-5. Distance methanogenic reactor level drops in relation to various volumes of sludge withdrawn at specified detention times	85

	Page
Table B-1. Location of data collected during each phase of the study	89
Table C-1. Data used in calculation of the gas production rates in each system	130
Table C-2. Summary of data used to calculate average values for influent feed COD during each phase of the study	131
Table C-3. Expanded data set for influent feed COD including average overall feed COD and standard deviation	132
Table D-1. Summary of cumulative COD fractions of average feed sample and reactor contents during raw feed study of each phase	134
Table D-2. Summary of cumulative COD fractions of average feed sample and reactor contents during homogenized feed study of each phase	135
Table D-3. Summary of incremental COD fractions of average feed sample and reactor contents during the primary sludge feed study of each phase	136
Table D-4. Summary of incremental COD fractions of average feed sample and reactor contents during homogenized feed study of each phase	137
Table G-1. Data used in calculation of the hydrolysis rate constants for the acidogenic reactor	146

LIST OF FIGURES

	Page
Figure 1. Electron flow through the three stages of methanogenesis (18)	6
Figure 2. Relationships between pH, bicarbonate alkalinity, and percent CO ₂ in anaerobic digester off-gas (20)	11
Figure 3. Effect of solids retention time (SRT) on percent removal of chemical oxygen demand (COD) (7)	16
Figure 4. Diagram of flow patterns in anaerobic reactor used in study	27
Figure 5. Diagram of gas handling apparatus	29
Figure 6. Incremental COD distributions for samples taken during phase III, 10-day HRT, during the primary sludge feed study, with equivalent particle size diameters of each fraction defined in Table 20 of Chapter V. The fraction between 0.001 and 0.04 micrometers represents the more soluble portion of each sample	60
Figure 7. Incremental COD distributions for samples taken during phase III, 10-day HRT, during the homogenized feed study, with equivalent particle size diameters of each fraction defined in Table 20 of Chapter V. The fraction between 0.001 and 0.04 micrometers represents the more soluble portion of each sample	62
Figure A-1. Feeding Apparatus including a) reactor waste valve, b) reactor feed valve, c) pump waste tube outlet, d) pump waste tube inlet, e) pump feed tube inlet, and f) pump feed tube waste line.	76
Figure F-1. Incremental COD distributions for samples taken during phase II, 20-day HRT, during the primary sludge feed study, with equivalent particle size diameters of each fraction defined in Table 20 of Chapter V. The fraction between 0.001 and 0.04 micrometers represents the more soluble portion of each sample	143

	Page
Figure F-2. Incremental COD distributions for samples taken during phase II, 20-day HRT, during the homogenized feed study, with equivalent particle size diameters of each fraction defined in Table 20 of Chapter V. The fraction between 0.001 and 0.04 micrometers represents the more soluble portion of each sample	144

LIST OF ABBREVIATIONS

g	gram
mL	milliliter
G	Relative Centrifugal Force
L	Liter
COD	Chemical Oxygen Demand, mg/L
HRT	Hydraulic Retention Time, days
SRT	Solids Retention Time, days
VS	Volatile Solids, %

ACKNOWLEDGEMENTS

I would like to thank Dr. Audrey D. Levine for her help and guidance throughout this project. Her patience and understanding are very much appreciated. A special thanks is also extended to Dr. Amur Chiang whose help allowed me to see this project through to its completion.

I wish to dedicate this thesis to my wife, Sandra, and to our first born, Adam. Sandra's love and devotion was never ending and made it very easy for me to continue. Adam's birth came midway through the research for this project and is what I believe to be my greatest accomplishment.

I love you Sandra.

I love you Adam.

CHAPTER I. INTRODUCTION

Anaerobic digestion is a biological waste treatment process that is used to convert organic wastes into gaseous end-products containing methane and carbon dioxide. The process occurs in three stages. Initially, complex organic compounds and particulate matter are hydrolyzed by facultative and anaerobic organisms. The end-products of hydrolysis undergo fermentation to form low molecular weight products such as acetic acid, hydrogen, and carbon dioxide. During the second stage, the products of the first stage are metabolized further to form acetate and hydrogen. In the final stage, acetate and hydrogen are converted biologically to methane and carbon dioxide.

A major application of anaerobic digestion processes is the stabilization of municipal wastewater sludges and industrial wastes. Typically, the methane gas produced by anaerobic digestion systems is used to fuel electric generators to provide an energy source for powering wastewater treatment plants.

The successful operation of anaerobic processes is dependent on maintaining a delicate balance among the different groups of anaerobic microorganisms by providing the proper growth conditions. Poor process performance is often attributed to an imbalance in the concentrations of the various microorganisms that are necessary for conversion of organic wastes. In some cases, successful operation has been achieved by separating the various stages of digestion. This process modification is referred to as a two-phase system, with hydrolysis and fermentation

occurring in the first phase and gas production in the second phase. The major benefits of phase separation include improved process stability and control. However, when using a two-phase system, interactions between various microbial species are limited. Because each phase produces end-products that are needed by the other, operational problems may arise.

The rate-limiting step of anaerobic digestion is considered by some to be the rate of hydrolysis of complex organic particles (9,10,28,29). Complete digestion can only occur when the waste is in soluble form. Increasing the hydrolysis rate can be accomplished by partially hydrolyzing the waste stream mechanically before it enters the anaerobic process (9,10). However, the effects of pretreatment of the waste are not well documented.

A better understanding of the effects of modifying the particle size distribution of the organic particulate components in the influent stream as related to energy recovery from anaerobic systems is needed. This research project was designed to investigate the effects of particle size reduction of a municipal primary sludge on the performance of anaerobic systems. Two anaerobic treatment systems were evaluated in this study, a conventional and a two-phase system.

CHAPTER II. OBJECTIVES AND SCOPE OF THE STUDY

The primary objective of this research project was to evaluate the effects that particle size reduction of influent primary sludge had on the performance of the anaerobic digestion process. Two treatment systems, a conventional and a two-phase anaerobic treatment system were operated simultaneously under identical conditions.

The effects of reducing the influent particle size on the operation of the two systems were evaluated by monitoring the following parameters:

1. Gas production,
2. Gas quality,
3. Volatile organic acids concentrations of reactor contents,
4. Chemical oxygen demand (COD) of influent and effluent waste streams,
5. COD of cumulative size fractions of samples of influent feed sludges and reactor contents, obtained by a fractionation technique involving sedimentation followed by centrifugation.

The reactors were operated as completely mixed anaerobic reactors and were maintained at approximately 35 degrees Celsius. The two-phase system consisted of two reactors connected through separate gas exit lines. A single reactor was used for the conventional system.

Substrate used in the study was primary sludge obtained from the Ames, Iowa, Water Pollution Control Plant. The substrate was collected on three separate occasions and stored in gallon jugs at 4 degrees Celsius until needed. The study was conducted in the Sanitary

Engineering Research Laboratory located in the Town Engineering Building at Iowa State University. The reactors were operated in a constant temperature room maintained at 35 degrees Celsius.

CHAPTER III. BACKGROUND

To provide background information, previous work on anaerobic processes with emphasis on process control and the various modes of operation is summarized. The use of anaerobic phase separation and studies researching the particle size distribution in anaerobic treatment systems are also discussed.

Anaerobic Degradation

The anaerobic process is the most widely accepted method for sludge stabilization. Anaerobic processes involve many facultative and strictly anaerobic organisms to bring about the complete conversion of complex mixtures of substrates to methane gas.

The process can be divided into three stages as shown in Figure 1 (14,18). In the first stage, insoluble, complex organic particulates are hydrolyzed by extracellular organisms into soluble substrates. Next, the soluble substrate is fermented by facultative and strictly anaerobic bacteria to produce long-chain organic acids, sugars, amino acids, and eventually smaller organic acids such as propionic, butyric, and valeric acid. Acetic acid, hydrogen, and carbon dioxide are also formed in the first stage (8,23). This hydrolysis step is believed by some to be the rate-limiting step in the overall process, although at high organic loadings, methanogenesis may gradually become rate-limiting (9,10,28,29). Bacteria involved in hydrolysis and subsequent fermentation have higher growth rates and are less sensitive to pH changes than the final stage methanogenic bacteria. Essentially no

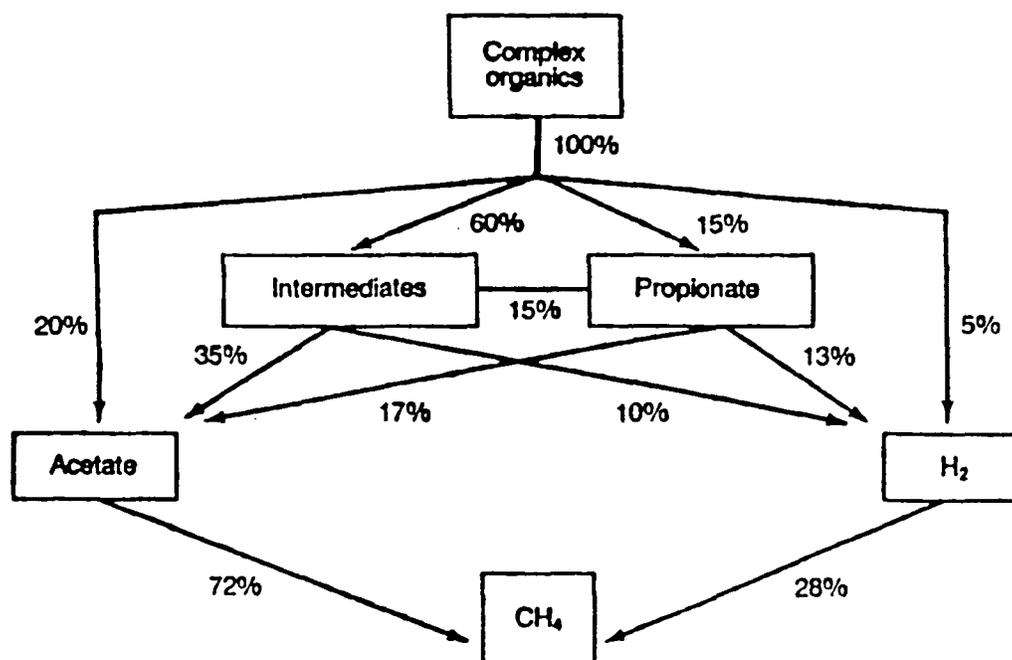


Figure 1. Electron flow through the three stages of methanogenesis (18)

waste stabilization occurs in the first stage, only transformation of the waste to a useable product for the second stage (10,23).

In the second stage, acetogenic bacteria form acetate and hydrogen by metabolization of the products from the first stage. These processes are referred to as acetogenesis and dehydrogenation, respectively. This step is essential for producing a substrate that can be utilized by the strictly anaerobic bacteria in the methanogenic stage.

Conversion of acetate and hydrogen to methane and carbon dioxide is the final stage in the anaerobic degradation process. The bacteria involved in the methanogenesis stage have slower growth and metabolic rates because the major portion of the energy is released as methane, leaving little energy for synthesis of new microorganisms. Therefore, the major portion of the degradable waste is converted to methane and carbon dioxide. Thus, methane production from waste conversion can be related directly to waste stabilization (19).

As shown in Figure 1, acetate conversion accounts for 72% of the methane based on the following relationship:

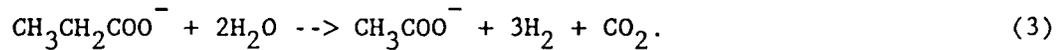


with the remaining 28% from H_2 utilization as follows:



The second equation is extremely important for efficient operation of the anaerobic process. Partial pressures of H_2 in the digesting sludge must be maintained at less than 0.000 1 atmospheres to allow for the

rapid conversion of fatty acids and their precursors as illustrated in the following equation for conversion of propionate to acetate (2,8,18,28,29):



As the partial pressure of H_2 increases, the conversion of fatty acids and H_2 production becomes rate-limiting. High partial pressures of hydrogen can develop and become a major concern in the treatment of complex wastes where as much as 30% of the electron flow is through propionate as shown in Figure 1 (18). However, when all three stages of anaerobic degradation are combined, the methanogenic organisms reduce H_2 to methane and maintain the required low H_2 partial pressures (28).

For effective waste stabilization to occur, a heterogeneous group of microorganisms must be present to conduct all stages of the anaerobic degradation process. A summary of the physical and chemical requirements for efficient anaerobic digestion to occur is given in Table 1 (20). Typically, for anaerobic treatment of primary sludge, sufficient nutrients are present. However, in the treatment of industrial wastes, nutrient supplements such as nitrogen and phosphorous may be required.

Complete waste stabilization results in the production of methane and carbon dioxide and each stage is critical to the success of the process. Imbalances may occur between the populations of methanogenic bacteria and the relatively fast growing acidogenic bacteria, resulting in a buildup of volatile acids. The increase in volatile acids

Table 1. Requirements for anaerobic digestion (20)

Temperature	
Mesophilic range	29 to 38 degrees Celsius
Thermophilic range	49 to 57 degrees Celsius
Anaerobic conditions	
Sufficient biological nutrients	
Nitrogen	
Phosphorous	
Others	
pH range	6.6 to 7.6
Bicarbonate alkalinity	2500 to 5000 mg/L as CaCO ₃
Absence of toxic materials	

concentration may be sufficient to cause a decrease in the pH buffering capacity, and subsequent process failure (10,20). Therefore, for efficient operation of anaerobic systems, proper control of pH and other operating parameters is very important. Several of the parameters indicating impending digester failure are listed in Table 2.

Process Control

The optimum pH for anaerobic digestion occurs between 7.0 and 7.2 with the normal operating range from 6.6 to 7.6 (20). Digester failure is usually evidenced by a decrease in pH caused by an increase in volatile acids concentration. However, the pH will not change until all

Table 2. Indicators of impending digester failure (20)

Parameters Increasing

Volatile Acids Concentration
CO₂ Percentage in Gas

Parameters Decreasing

pH
Total Gas Production
Waste Stabilization
Alkalinity

the buffering capacity of the system is used up and the system is at failure. Therefore, of the many parameters, concentration of volatile acids is the most useful in detecting operational problems.

Changes in pH are buffered by the carbon dioxide-bicarbonate system. The relationships between pH, bicarbonate alkalinity, and percent CO₂ in the off-gas are shown in Figure 2 (20). As shown in the figure, a bicarbonate alkalinity of 2500 to 5000 mg/L provides adequate buffer capacity to allow the volatile acids concentration to increase substantially before the pH drops below the normal operating range.

If the volatile acids concentration continues to increase and reduce the bicarbonate alkalinity below the limit shown in Figure 2, then the bicarbonate alkalinity should be controlled. With proper pH adjustment, volatile acids concentrations of up to 6000 mg/L can be tolerated without loss in methane production (23). One method for controlling pH in a digester is the addition of sodium bicarbonate (20).

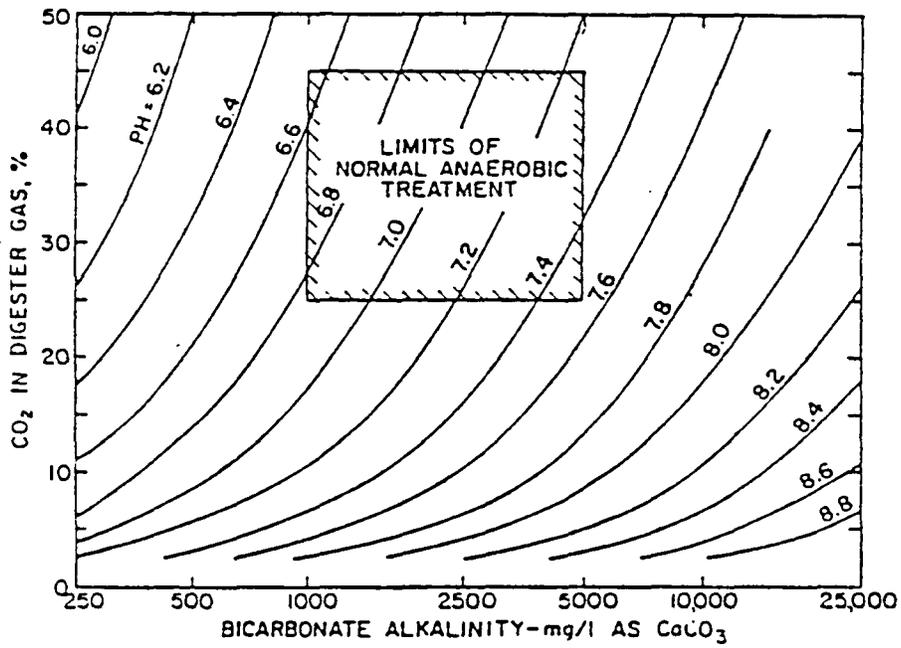


Figure 2. Relationships between pH, bicarbonate alkalinity, and percent CO₂ in anaerobic digester off-gas (20)

Sodium bicarbonate can be added to the digester to raise the alkalinity and resulting pH until the methanogenic bacteria are able to efficiently degrade the volatile acids. Toxicity of sodium bicarbonate is not a problem as long as the bicarbonate alkalinity in the digester remains below 5000 mg/L.

Toxicity is of major concern in microbial systems. The toxicity of known substances is usually broken down into two categories, organic and inorganic substances. The molecular structures of organic substances have been directly correlated to toxicity (23,29). Chloro substitution, aldehydes, double bonds and benzene rings have been shown to exhibit toxicity while the addition of hydroxyl groups and increased carbon chain length will tend to decrease toxicity. Inorganic substances have also been reported to cause toxicity and Table 3 contains a summary of the toxic concentrations of select inorganics from a wide group which is made up of alkali and alkaline-earth metals, heavy metals, ammonia-nitrogen, and sulfide (23).

Ammonia-nitrogen is generally accepted to be inhibitory at concentrations between 1500 and 3000 mg/L and toxic at concentrations above 3000 mg/L (23). Ammonia-nitrogen is the total nitrogen existing as ammonium ion and ammonia gas based on the following equilibrium relationship:



The fraction of ammonia in the gaseous phase increases with pH and concentrations above 100 mg/L may cause severe toxicity (23). Soluble

Table 3. Concentrations of inorganics reported to be toxic to anaerobic digestion (23)

Inorganic Substance	Toxic Concentraions, mg/L	
	Moderately toxic	Strongly toxic
Sodium	3,500-5,500	8,000
Potassium	2,500-4,500	12,000
Calcium	2,500-4,500	8,000
Magnesium	1,000-1,500	3,000
Ammonia-nitrogen	1,500-3,000	3,000
Sulfide	200	200
Copper	---	0.5(soluble)
Chromium VI	---	3.0(soluble)
Chromium III	---	2.0(soluble)
Nickel	---	2.0(soluble)
Zinc	---	1.0(soluble)

sulfides at concentrations greater than 200 mg/L are inhibitory, and heavy metals show toxicity at soluble concentrations ranging from 0.1 to 3 mg/L (23,28). It should be noted that substances must be soluble to have toxic effects; therefore, heavy metals may be precipitated with sulfide and reduce both the heavy metal and soluble sulfide concentrations to below toxic levels depending on the pH.

Modes of Operation

Since the introduction of the conventional unmixed, unheated anaerobic digester for treatment of wastewater sludges, many developments have been made to increase process efficiency. More recent design practices include the use of a completely mixed, heated, high rate system. The high rate system operates at or above 35 degrees Celsius, the optimum temperature for mesophilic methanogenic bacteria. Hydraulic retention times (HRTs) are 10 to 20 days, as compared with 30 to 60 days for the conventional system. The minimum HRT of 10 days corresponds to the minimum solids retention time (SRT) or minimum regeneration time of the slow growing methanogenic organisms (7). The equations defining hydraulic retention time (HRT) and solids retention time (SRT) are:

$$\text{HRT} = \frac{V}{Q} \quad (4)$$

$$\text{SRT} = \frac{V * X_R}{Q * X_W} \quad (5)$$

where:

V = Reactor volume;

Q = Digested sludge wastage rate;

X_R = Concentration of reactor contents;

X_W = Concentration of wasted digested solids.

For completely mixed, high-rate digesters, the concentration of the

reactor contents (X_R) is equal to the concentration of the wasted digested sludge (X_W), thereby making the SRT equal to the HRT (7). If the SRT is allowed to decrease below the minimum regeneration time of 10 days, gas production and removal of degradable organic compounds will decrease dramatically as shown in Figure 3 (7).

Increasing SRT without increasing HRT is beneficial because high treatment efficiency can be obtained with smaller, less expensive digesters. Also, economical anaerobic digestion of dilute wastes, which require increased hydraulic loading of the systems, is possible using an increased SRT. One of the first attempts at separating SRT from HRT was the development of the anaerobic activated sludge system, with more recent design modifications including fixed film/filter, expanded/fluidized bed, upflow anaerobic sludge blanket and baffled systems(7,18,32). Each of the systems is capable of maintaining a large bacterial mass within the system to accomplish high waste stabilization rates per unit volume.

High-rate digesters, and digesters able to separate SRT from HRT, are capable of increasing the total process efficiency and stability of the anaerobic system. Process efficiency and stability can also be improved by increasing the efficiency of the individual stages in the process. Phase separation allows the acidogenic organisms to be separated from the methanogenic organisms, thus offering an opportunity to optimize each phase separately (5,9,10,23).

Another approach for improving overall process efficiency and stability is to optimize conditions for the rate-limiting step of the

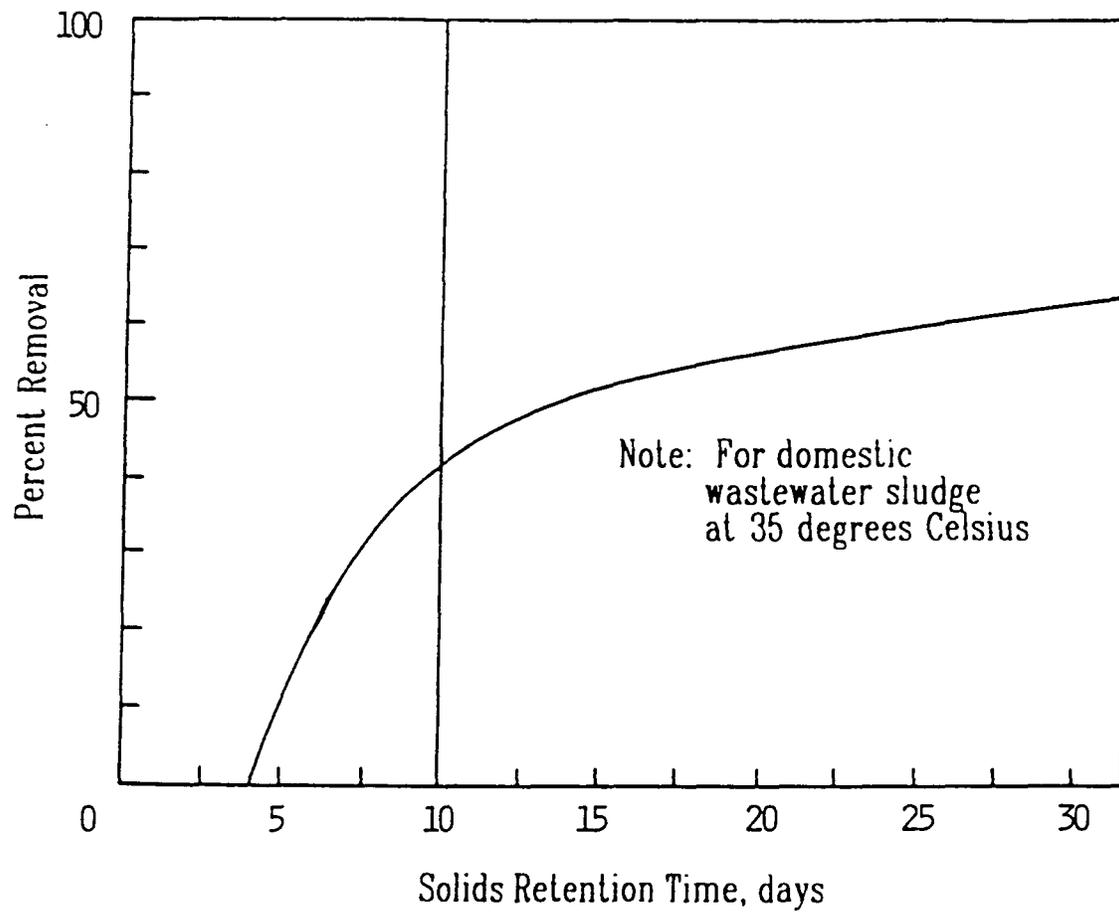


Figure 3. Effect of solids retention time (SRT) on percent removal of chemical oxygen demand (COD) (7)

degradation process. In cases where the sludge to be processed contains high concentrations of solids, the hydrolysis step may be rate limiting. An option for increasing the overall hydrolysis rate may be to mechanically modify the particle size distribution of the influent waste stream.

High Rate Digestion

High rate digesters consist of a conventional reactor that is completely mixed and heated to a temperature of 35 degrees Celsius or above. The significant feature of high rate digesters is that considerably higher organic loads can be applied at HRTs well below those characteristic of conventional unmixed, unheated digesters. Modern high rate digestion systems usually consist of a two-stage system; the first stage performs the treatment and the second stage is used to thicken and store the digested sludge. The second stage is usually unheated and unmixed and produces a relatively clear supernatant. Design criteria for high rate digestion systems vary widely. Typical loading rates and detention times are given in Table 4 (31,36). Significant results from several laboratory studies that have been performed using high rate anaerobic systems are given in Table 5. As shown in Table 5, the methane production rate based on grams of COD destroyed is very close to the theoretical value of $0.35 \text{ L CH}_4/\text{g COD}$ destroyed for each of the systems, assuming conversion of all data to a standard temperature of 0 degrees Celsius. The theoretical value for

Table 4. Design criteria for high rate anaerobic digestion systems

	Ref 31	Ref 36
Solids retention time (days)	10-20	10-15
Loading rate (g VS/L/day)	2.4-6.4	1.6-3.2
Loading rate (g COD/L/day) ^a	4.8-12.8	3.2-6.4

^aBased on approximate value of 0.5 g VS/g COD (4,33).

methane production per gram of COD destroyed is based on the following equation:



Therefore, the volume of methane per gram of COD at a standard temperature and pressure of 0 degrees Celsius and 1 atmosphere can be calculated as:

$$\begin{aligned} \text{Volume CH}_4 &= \frac{(1 \text{ gram COD destroyed})(22.4 \text{ l/mole})}{2(32 \text{ g O}_2/\text{mole O}_2)} \\ &= 0.35 \text{ L CH}_4/\text{gram COD destroyed.} \end{aligned}$$

Phase Separation

Historically, anaerobic digester design has been based on providing an environment in which both the acidogenic and the methanogenic organisms could survive simultaneously. Digesters have been designed to promote the growth of the sensitive and slow growing methanogenic

Table 5. Summary of high rate anaerobic treatment systems operating in the approximate range of 10 to 20 days HRT

	10	10	11	11	14	15	20	23 ^a
Hydraulic Retention Time (HRT), days								
VS Loading, g VS/L/d	3.73	2.70	2.19	3.35	2.79	1.80	1.29	1.67
Total Gas Produced, L/g VS destroyed	1.12	1.28	1.20	1.20	1.20	1.30	1.28	1.02
Methane Percentage, %	65 ^b	56.8	65 ^b	65 ^b	65 ^b	57.6	57.2	66
Methane Production Rate, L CH ₄ /g VS destroyed	0.73	0.73	0.78	0.78	0.78	0.75	0.73	0.67
COD Loading ^a , g COD/L/d	7.46	5.40	4.38	6.70	5.58	3.60	2.58	3.34
Methane Production Rate ^c , L CH ₄ /g COD destroyed	0.37	0.37	0.39	0.39	0.39	0.38	0.37	0.34
Theoretical Methane Production Rate, L CH ₄ /g COD destroyed	0.35	0.35	0.35	0.35	0.35	0.35	0.35	0.35
Reference	21	26	25	25	25	26	26	27 ^b

^a Full scale digester.

^b Assumed percentage of methane in gas.

^c Based on approximate value of 0.5 g VS/g COD (4,33).

organisms without regard to controlling growth of the much less sensitive and rapidly growing acidogenic organisms. Recognizing that the acid formers are not in an optimum environment, some researchers have proposed controlling anaerobic degradation by phase separation of the acidogenic and methanogenic organisms, thereby creating optimum growth environments for each group of organisms (4,5,6,9,10,23).

Two-phase digestion involves the use of two completely mixed digesters in series, one for hydrolysis and acidification of the waste by the acidogens, and the other for methane production. The environment in the acidogenic digester allows for hydrolysis of insoluble organic matter, reduction of the nitrogen and oxygen contents, and a pH below 7.0 (5,10). Production of volatile organic acids in the acidogenic reactor provides the methanogenic reactor with an easily digestible substrate that can be efficiently converted to gaseous end products.

Complete phase separation is not recommended in the anaerobic process because of problems that may arise from the lack of interaction among the organisms (10,23). The gas in the acidogenic digester typically contains a high concentration of hydrogen that is utilized by the methanogenic organisms. If this gas is not transferred to the methanogenic digester, the conversion of longer chain intermediates to acetic acid could be inhibited. Also, if the hydrolysis step is not rate-limiting, phase separation would cause a buildup of volatile acids in the acidogenic digester to concentrations inhibitory for methanogenesis.

Improved digester stability and process control are the major

advantages of phase separation (9,10,23). The use of two-phase systems is ideal for influent feeds that are highly variable in quantity or quality. The series configuration of the two-phase system also provides additional buffer capacity for protecting the sensitive methanogens against exposure to slug loads of inhibitory materials (13). When utilized in the proper environment, phase separation can have a substantial impact on the degradation of waste. Ghosh et al. (10) reported operating a two-phase digester treating waste activated sludge at one-half the detention time of a high-rate digester while exhibiting higher rates of solids stabilization and methane production.

Particle Size

Anaerobic degradation involves the breakdown of large particles into smaller particles that can be consumed by cellular organisms and converted to a gaseous form. The process by which the larger particles are broken down is the result of first stage enzymatic hydrolysis. Extracellular organisms secrete enzymes that interact with the surface of the large particles causing breakdown to smaller particles. The process continues until the particles are small enough to be metabolized by the acidogenic bacteria. As the particles are reduced in size during treatment, the available surface area is increased and therefore, the apparent hydrolysis rate is increased.

Several investigators have studied the hydrolytic process and found that the rate of degradation is directly related to the available surface area of the particles in the waste stream (1,9,15,16,17,24). As the effective particle size of the waste particles decreases, the

surface area increases. It is possible that the effective particle size distribution of a waste stream may be modified to a range where hydrolysis is not rate-limiting.

The fact that smaller particles were hydrolyzed at a much greater rate than larger particles was demonstrated in a study that employed phase separation (15). An increase in surface area occurred mainly in the smaller particle size fractions of the digesting sludge, and the rate of hydrolysis for the small particles was greater than that of the large particles.

Modeling efforts focused on the acidogenic phase of anaerobic degradation, including both hydrolysis and fermentation, have been limited. However, a working model was developed by Eastman and Ferguson and is given below:

$$(F_o - F) + (S_o - S) + (X_o - X) + (P_o - P) = 0 \quad (8)$$

where:

$(F_o - F)$ = change in concentration of degradable particulate COD;

$(S_o - S)$ = change in concentration of substrate COD;

$(X_o - X)$ = change in concentration of viable biomass COD;

$(P_o - P)$ = change in concentration of product COD (9).

As shown in equation 8, there is no change in the overall COD of the anaerobic system during acidogenesis, only a transformation of the COD between phases.

The rate of hydrolysis enters into equation 8 in calculation of the

change in the degradable particulate COD. Eastman and Ferguson stated that at constant temperature and pH, the hydrolysis rate is approximately first-order with respect to the remaining concentration of degradable particulate COD as shown in the following equation:

$$R_h = k_h F \quad (9)$$

where:

k_h = first-order hydrolysis rate constant;

F = concentration of degradable particulate COD (9).

As stated previously, increasing the hydrolysis rate can have a major effect on the rate of degradation. The immediate effect that increasing the hydrolysis rate constant would have on the particulate COD component of equation 8 can be seen by the following equation for a completely mixed continuous flow reactor operating at steady state:

$$F = \frac{F_o}{1 + k_h \theta} \quad (10)$$

where:

F_o = influent concentration of degradable particulate COD;

F = effluent concentration of degradable particulate COD;

θ = hydraulic retention time of the system (9).

A decrease in particulate COD would not be completely related to an increase in the hydrolysis rate, as evidenced in equation 10. Cell growth would contribute to a decrease in the effluent particulate COD

concentration. However, an increase in the hydrolysis rate constant should correspond to a decrease in the effluent concentration of degradable particulate COD, as shown in equation 10. Higher rate constants cause an increase in the change in degradable particulate COD in equation 8 for a given HRT and SRT. As a result, the concentration of soluble COD available for metabolism increases. Any transformation of particulate COD to soluble or product COD will result in improved process performance. Therefore, reduction of the particle size distribution of the influent waste stream to increase the rate at which the waste is converted to a readily degradable substrate may be beneficial for improving waste treatability.

The literature related to operation of anaerobic treatment systems was used as a basis for this experimental study. The study investigated the effects of particle size reduction of the particulate components in a municipal primary sludge on the performance of the two separate anaerobic systems, a conventional system and a system utilizing phase separation.

CHAPTER IV. EXPERIMENTAL STUDY

The basis of the experimental study was to evaluate the effects of particle size reduction of the feed substrate on the performance of anaerobic digesters. A comparison was made between two anaerobic systems, conventional and two-phase. The conventional reactor consisted of a single, completely mixed anaerobic reactor while the two-phase system employed separate completely mixed anaerobic reactors for acidogenesis and methanogenesis.

Both anaerobic systems were operated at three different hydraulic retention times (HRTs) or phases. During each of the three phases, the effects of particle size reduction of the feed substrate were studied. Each phase was divided into two sub phases based on the physical characteristics of the feed sludge. Raw primary sludge was fed to each system during one sub phase and homogenized primary sludge during the other. Both systems were monitored on a regular basis to collect performance data.

Experimental Design

Completely mixed anaerobic reactors maintained at 35 degrees Celsius were used in the experimental study. A gas handling apparatus was connected to each system to allow for feeding and wasting of feed substrate, sampling of the off-gas for analysis, and measurement of the total gas production.

Reactor Design

Three identical plexiglas reactors were operated as completely mixed anaerobic reactors. The major features of the reactors are shown in Figure 4. Design of the reactors was based on studies conducted by Dague et al. (6) and design parameters from Holland and Chapman (12). A list of design parameters is presented in Table 6.

Table 6. Summary of reactor design parameters from Holland and Chapman (12)

-
- (1) Digested liquid height equal to reactor diameter
 - (2) Propeller diameter equal to one-third the tank diameter
 - (3) Propeller height from the reactor bottom equal to propeller diameter
 - (4) Four baffles vertically mounted at the reactor wall and extending from the reactor bottom to above the liquid surface
 - (5) Baffle width equal to one-tenth the reactor diameter
-

Each anaerobic reactor had an inside diameter of 15.24 cm and a height of 26.67 cm. The thickness of the plexiglas was 1 cm. The reactor volume was 4.865 L with a 4 L digested sludge volume. A flanged connection at the top of each reactor allowed easy access into the reactor, and an O-ring provided an air-tight seal. Ball valves, 0.95 cm in size, were located at distances of 5.1 and 12.7 cm from the bottom of the reactor and used for sludge feeding and wasting.

The two-phase system consisted of two identical reactors connected by joining their separate gas exit lines to exit through a combined gas

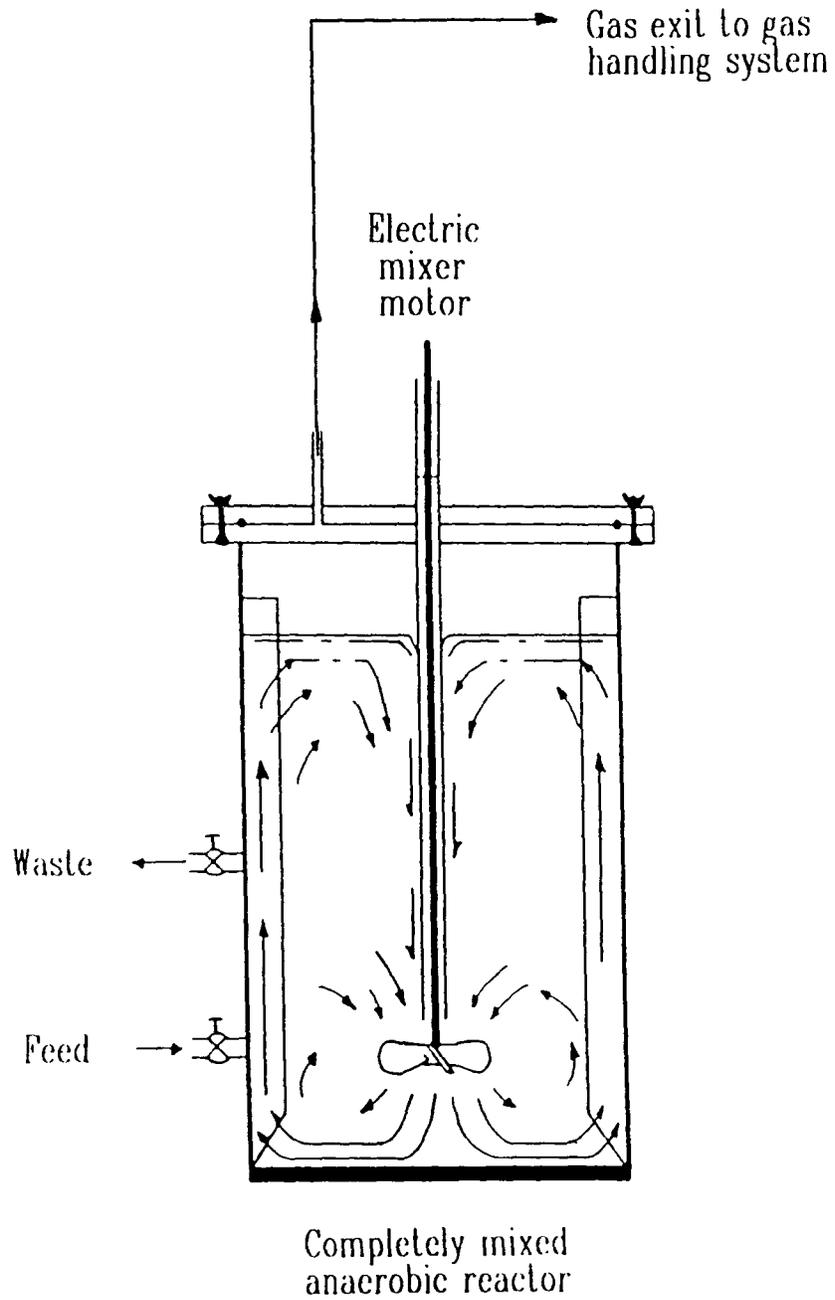


Figure 4. Diagram of flow patterns in anaerobic reactor used in study

handling apparatus. The conventional system consisted of a single reactor and gas handling apparatus.

Temperature and Mixing

The reactors were operated at approximately 35 degrees Celsius. High pressure steam provided the primary source of heat in an enclosed room. In addition, thermostatically controlled electric heaters were used during interruptions of the steam heat.

The contents of each reactor were completely mixed using a 3-blade marine type propeller attached to a variable speed mixer. The propeller blade was approximately 4 cm in diameter, and the shaft was encased in a stainless steel tube as shown in Figure 4. The tube was submerged in the digested liquid to ensure anaerobic conditions within the reactor. Stainless steel baffles were added to each reactor to achieve an axial flow mixing pattern in the reactor, also shown in Figure 4.

Mixing of the reactors was accomplished by operating the mixers at approximately 1500 rpm for 7.5 minutes every 30 minutes. This procedure was used because continuous operation of the mixers resulted in an increase in reactor temperature.

Gas Handling

Gas exited each of the two experimental reactor systems to separate gas handling systems as shown in Figure 5. The water lock device used consisted of two interconnected 500 mL bottles that protected the remainder of the gas handling apparatus from suction conditions while feeding and wasting from the reactors. Each bottle contained

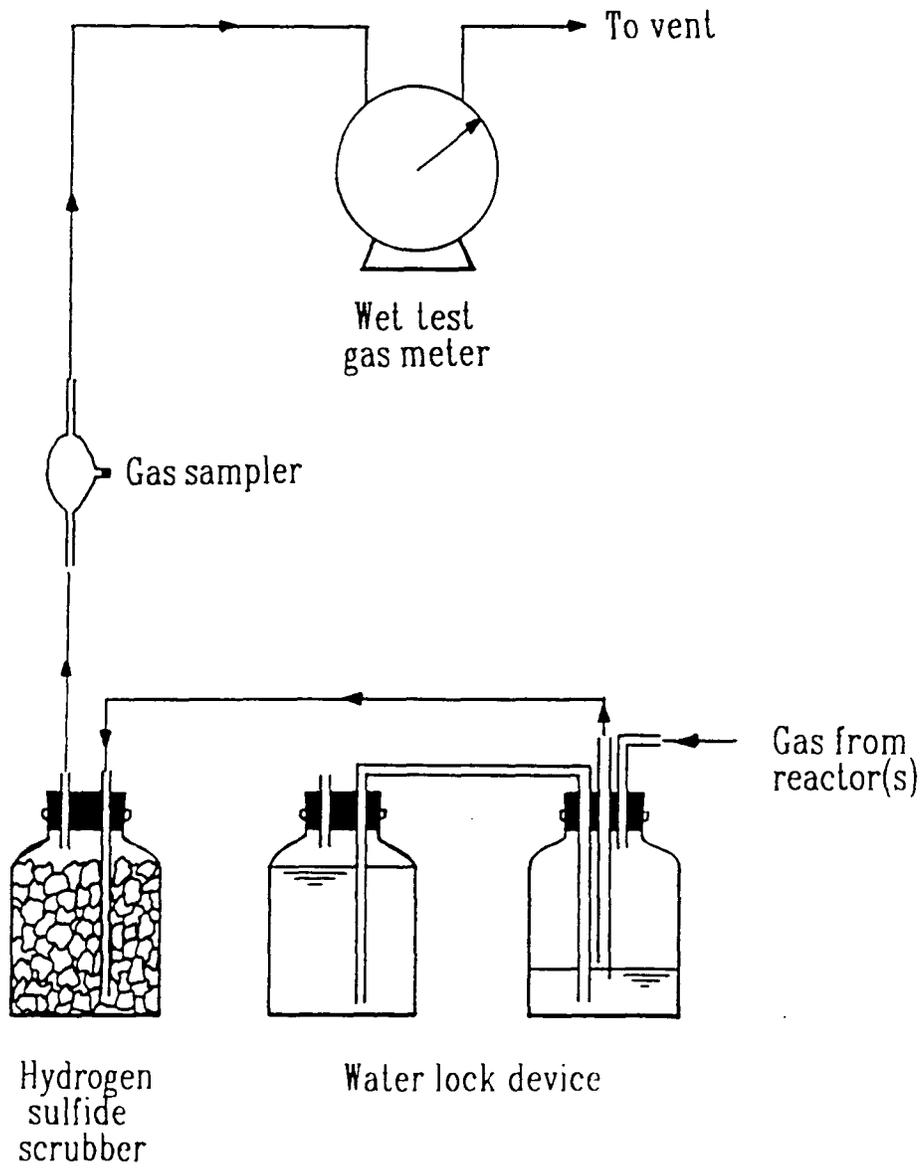


Figure 5. Diagram of gas handling apparatus

approximately 250 mL water acidified to a pH of 1 with sulfuric acid.

A water bridge connected the two bottles to prevent back mixing of gas and to monitor potential leaks in the system. As gas was produced in the reactors, the gas pressure in the reactor head space increased. The increase in gas pressure caused a transfer of water from the first bottle to the second. Transfer of water continued until the level of water in the first bottle reached the bottom of the middle gas exit tube and a portion of the gas was released from the water lock. The water levels in the water lock during gas release are as shown in Figure 5. While the reactors were operating under gas-tight conditions, a height difference in the two water levels existed. The difference was due to the position of the gas exit tube from the system. Gas could not exit the system until the level of water in the first bottle reached the bottom of the gas exit tube. The difference in water levels also provided a visual confirmation that the system was air tight. If a gas leak occurred in the system, the water levels in both bottles were identical.

The water lock was an essential component of the feeding process. As sludge was withdrawn from the reactors during feeding, a negative pressure developed in the first bottle of the water lock and resulted in a transfer of water from the second bottle. Simultaneous feeding and wasting was continued until the appropriate amount of sludge had been wasted. Wasting was then halted and feeding resumed until the level of sludge in the reactor had risen to the full mark with a corresponding transfer of water from the first bottle back to the second. After

feeding, the water levels in the water lock would restabilize with the first bottle having a lower level than the second, as shown in Figure 5.

Gas flow from the reactors was through the water lock then through a hydrogen sulfide gas scrubber. The scrubber consisted of ferric oxide coated wood shavings packed in a stoppered 500 mL glass bottle.

Hydrogen sulfide gas had to be removed from the gas stream to prevent release of toxic gases into the room and corrosive damage to the gas meter. Following the scrubber, the gas exited through a 50 mL gas sampler. The total gas produced was monitored using a GCA Precision Scientific-Wet Test Gas Meter. The 50 mL gas sampler was used to collect samples for analysis of methane and carbon dioxide content.

System Operation

During each phase of the project, the effects of reducing the particle size of the feed on the performance of the two anaerobic systems were studied. Comparisons were made between the two systems during each phase. Gas production data were compiled for both systems while other operating parameters were studied for the feed and effluent from each reactor. The parameters that were monitored are listed in Table 7. The concentrations of individual volatile acids were monitored to characterize the effect of changing feed sludge and HRT on reactor performance. The contents of each reactor and a sample of the feed sludge were fractionated into four size fractions by sedimentation followed by centrifugation at the conclusion of each study, and the chemical oxygen demand (COD) of each fraction analyzed. Each size fraction included particles with an equivalent diameter less than or

Table 7. Operating parameters analyzed for feed and effluent from each system

(1)	Temperature
(2)	pH
(3)	Chemical oxygen demand (COD)
	a) Total
	b) Centrate of sample centrifuged at 39100 G
	c) Total COD and COD of the centrate of samples after fractionation by sedimentation and centrifugation at the conclusion of each study
(4)	Alkalinity
(5)	Individual volatile organic acids
(6)	Percentage of methane and carbon dioxide in the off-gas from each system

equal to the smallest particle removed, as calculated using the Stokes' equation. The concentration of COD associated with each fraction was then calculated and the size distributions of COD compared for individual reactors during different modes of operation.

Hydraulic Retention Time

The three HRT values chosen for the study were 10, 15, and 20 days. The values for HRT during the three phases and the length of time both systems were fed each of the two feed sludges are given in Table 8. The systems were run as conventional high-rate digesters, thereby making the HRT equal to the solids retention time (SRT). The detention time for

phase I was fifteen days, phase II twenty days, and phase III, the minimum SRT of ten days (1). The acidogenic reactor of the two-phase system was operated at a one day HRT throughout the study.

During phase I, the conventional reactor and the methanogenic reactor were operated at a fifteen day detention time. Thus, the two-phase system had a six (6) percent longer solids retention time (SRT) due to the one day HRT of the acidogenic reactor. During phases II and III, the methanogenic reactor had an HRT of nineteen and nine days respectively to provide the same total HRT as the conventional system.

Reactor Start Up

After assembling each of the anaerobic systems, all three anaerobic reactors were completely filled with effluent from one of the secondary digesters at the Water Pollution Control Plant in Marshalltown, Iowa. The digesters were not mixed for approximately 18 hours to prevent air in the gas head space from being mixed throughout the reactor. During the initial time period, gas formed in the reactor and displaced the air occupying the gas head space. After 18 hours, mixing of the reactors and the batch feeding program was started.

Feeding Sequence

All three reactors were fed substrate on a batch basis. The methanogenic reactor (reactor no. 2) and the conventional reactor (reactor no. 3) were fed once every 24 hours. Since the acidogenic reactor (reactor no. 1) was operated on a 1 day HRT, it was fed every 8 hours to minimize shock loading. Detailed daily reactor feeding

Table 8. Values for SRT of each system and length of individual studies for each phase of the experimental study

Phase of Study	HRT, days	Length of Phase, days	Length of each subphase, days	
			Primary Sludge feed subphase	Homogenized feed subphase
Phase I. 15-day HRT		40 ^a	16	24
Conventional System	15			
Two-phase System				
Acidogenic	1			
Methanogenic	15			
Phase II. 20-day HRT		44	16	28
Conventional System	20			
Two-phase System				
Acidogenic	1			
Methanogenic	19			
Phase III. 10-day HRT		32	21	11
Conventional System	10			
Two-phase System				
Acidogenic	1			
Methanogenic	9			

^a Had been run at 15-day HRT for 45 days prior to collecting data for phase I.

procedures for all three reactors are given in Appendix A.

As noted in Table 8, both systems had been operating at the fifteen day HRT for 45 days before phase I was begun. After 16 days of feeding the raw primary sludge to each system, the feed substrate was switched to homogenized primary sludge. In phase II, the feeding sequence was reversed from that of phase I and the homogenized feed was fed first. Both systems were allowed to acclimate to the twenty day HRT before initiating the raw feed study. The feeding sequence was reversed once again in phase III, beginning with raw primary sludge used as feed substrate, followed by the homogenized feed study.

A major consideration in comparing the performance of the two treatment systems was determining if a pseudo steady state condition existed. Upon changing the HRT of the systems, sufficient time had to be allowed for acclimation before any meaningful data could be obtained. Each system was first fed at the new HRT for a time at least equal to the HRT. Feeding was then continued until the daily gas production rates, as shown in the laboratory data in Appendix B, reached a relatively steady-state value. Determination of the steady-state condition was accomplished by calculating five day averages and standard deviations of the daily gas production rates. Upon achieving an average gas production rate with a coefficient of variation of less than or equal to ten percent, the next study was begun. The coefficient of variation as a percent was calculated by dividing the standard deviation by the average gas production rate and multiplying by 100.

Feed Substrate

The feed substrate used throughout the study was primary sludge that was obtained from the Ames, IA, Water Pollution Control Plant. The reactors were operated at three different hydraulic retention times (HRTs). Prior to beginning each phase of the study, approximately 40 gallons of sludge were collected. Data on the characteristics of the feed sludge are presented in Table 9.

The sludge obtained for feeding was stored in one-gallon containers at 4 degrees Celsius until needed. This temperature was closely monitored to prevent the possible changes in particle size distributions of the sludge due to freezing. Prior to feeding, the sludge was warmed to approximately 35 degrees Celsius in the room overnight.

Two separate studies were conducted during each phase, one where the primary sludge was fed "as received" and the other where the sludge was homogenized by blending prior to feeding. Therefore, after the sludge was warmed overnight, it was either poured directly into the feed jug or mechanically blended before being poured into the feed jug depending on the mode of operation.

Blending of the sludge during the 15 day HRT study was accomplished by use of a common household blender. However, due to excessive wear, the blender became unuseable and had to be replaced. Subsequently, a high speed laboratory homogenizer was used to blend the sludge.

Analytical Techniques

Process control of each reactor was accomplished by measuring various parameters of both the feed sludge and reactor contents. A list

Table 9. Characteristics of feed sludge

HRT	Parameter	Range	Average
10-day ^a	pH	5.5 - 5.7	5.6
	Alkalinity (mg/L)	1613 - 2335	1974
	COD (g/L)		
	As received	49.5	49.5
	Homogenized ^b	38.8 - 42.3	41.1
	Total Solids (%) ^b	3.56	3.56
15-day ^c	pH	5.5 - 5.9	5.7
	Alkalinity (mg/L)	1260 - 1875	1567
	COD (g/L)		
	As received	32.7 - 45.8	39.7
	Homogenized ^b	30.6 - 51.8	38.0
	Total Solids (%) ^b	2.71	2.71
20-day ^d	pH	5.2 - 6.0	5.6
	Alkalinity (mg/L)	1580 - 1850	1715
	COD (g/L)		
	As received	27.6 - 51.8	37.9
	Homogenized ^b	32.7 - 46.6	39.4
	Total Solids (%) ^b	2.99	2.99

^aSludge collected on Nov. 30, 1986.

^bValues for total solids percentage were obtained from Ames Water Pollution Control Plant.

^cSludge collected on Sept. 8, 1986.

^dSludge collected on Oct. 16, 1986.

of the analyses performed and the frequency of measurement is given in Table 10. Daily measurements included determinations of pH and temperature. Other analyses were performed less frequently and included chemical oxygen demand (COD), alkalinity, individual volatile organic acids, and gas composition. Each of the analyses is described briefly

below.

pH

The pH of the feed sludge and the reactor contents were measured using a Beckman Expandomatic IV pH meter. The meter was standardized with pH 4.00 ± 0.01 and 7.00 ± 0.01 buffers prior to use. The standardized meter was also used in the measurement of alkalinity.

Alkalinity

Analysis of the total alkalinity of both the feed sludge and the reactor contents were determined immediately after the pH determinations using the potentiometric titration method described in part 403, procedure 4c of Standard Methods (30). The sample size was 25 mL and the final end point was at pH equal to 4.5.

Temperature

The temperature of the reactor contents was measured using a mercury thermometer. A mercury thermometer submerged in water was also used to monitor the ambient air temperature. The thermometer was submerged to minimize effects of minor temperature fluctuations caused by opening and closing the door to the room.

COD

The chemical oxygen demand (COD) of the feed sludge and the reactor contents was determined using a modified method based on the reflux, colorimetric method for determination of chemical oxygen demand (COD) described in part 508c of Standard Methods (30). Actual analysis was

Table 10. Summary of analyses performed and frequency of measurement

Parameter	Frequency of measurement
pH	daily
Temperature	daily
COD	beginning and end of each sub phase; otherwise weekly
Alkalinity	same as COD
Volatile Acids	same as COD
Gas Composition	weekly

based on a method developed by Zimpro, Inc., ZP-166. Variations from the standard method included using a 0.7 N $K_2Cr_2O_7$ digestion solution and a sulfuric acid reagent made by dissolving 29.5 g $AgSO_4$ in 4.08 kg concentrated H_2SO_4 . The digestion vessels used were 16 x 125 mm culture tubes with TFE-lined screwcaps. Sample volume was 5.0 mL and reagent quantities added included 1.5 mL digestion solution and 6.5 mL sulfuric acid reagent. The maximum COD concentration per vessel was calculated as 1680 mg/L. Samples were diluted to contain between 200 and 1400 mg/L COD per 5 mL sample. The absorbance of digested samples was measured at 600 nm using a Beckman Spectrophotometer with 10 mm cells.

Weekly COD determinations for individual reactor contents and feed sludges included calculation of the total COD and the COD of the centrate of a sample centrifuged at a relative centrifugal force of

39100 G. The relative centrifugal force, G , when multiplied by the acceleration due to gravity, g , results in the radial acceleration of the centrifuge. The radial acceleration was also used in calculating the equivalent spherical diameter of the smallest particle removed at various G values. The centrate after centrifugation at 39100 G was assumed to consist of the soluble portion of the sample.

At the conclusion of the 10-day and 20-day HRT studies, contents of each reactor and a sample of the feed sludge were fractionated into three size fractions. The COD of each fraction and the total COD of the treated sludge were analyzed. The same fractionation technique was used for all samples and involved the use of sedimentation followed by centrifugation at two different speeds. The fractionation procedure for analysis of the size distribution of the samples from each reactor and the feed sludge sample was conducted as follows:

1. A 200 mL sample was diluted to 2 L in a graduated cylinder,
2. The graduated cylinder was capped and the contents thoroughly mixed by inverting the cylinder twenty times,
3. One liter of the diluted sample was then transferred to a standard one (1) liter Imhoff cone and allowed to settle for 30 minutes under quiescent conditions,
4. After 30 min., 50 mL of supernatant out of the top one-third of the Imhoff cone was transferred to a 100 mL volumetric flask, and another 50 mL of the supernatant transferred to a 50 mL high-speed centrifuge tube, 2.6 cm in diameter and 10 cm long, for further fractionation,
5. The supernatant in the volumetric flask was diluted to 100 mL, mixed thoroughly, and separate 5 mL samples transferred to a 5 mL graduated cylinder using a broken tipped pipette and then to the COD culture tubes for analysis,
6. The 50 mL sample in the centrifuge tube was first centrifuged

at a relative centrifugal force of 1935 G for 30 min.,

7. At the end of the 30 min. centrifugation, duplicate 5 mL samples of the centrate in the centrifuge tube were transferred to the COD culture tubes for analysis,
8. The sample was then centrifuged at a relative centrifugal force of 39100 G for 30 min.,
9. Duplicate 5 mL samples of the centrate after centrifugation at 39100 G were analyzed for COD content.

The fractionation process described above resulted in a distribution of three size fractions for each sample. Each fraction along with the total COD of each sample was divided into separate groups as described in Table 11. Equivalent spherical diameters of the smallest particle removed in each step of fractionation were then calculated using Stokes' equation for sedimentation.

Stokes' equation is based on the assumption that particles falling through a viscous medium quickly attain a terminal velocity and the parameters of the suspending medium and those of the particle are related as follows:

$$V = \frac{2 g d^2 (p_1 - p_2)}{9\eta} \quad (11)$$

where:

V = particles terminal velocity;

g = acceleration due to gravity;

d = diameter of equivalent sphere;

p_1 = density of the particle;

p_2 = density of the suspending medium;

Table 11. Summary of individual COD fractions

Group	Fractionation technique
Total	Total COD of sample before fractionation
I	COD of supernatant from Imhoff cone after the diluted sample was allowed to settle for 30 min.
II	COD of centrate after supernatant from Imhoff cone had been centrifuged at 1935 G for 30 min.
III	COD of centrate after supernatant from Imhoff cone had been centrifuged at 39100 G for 30 min.

n = viscosity of the suspending medium.

When a centrifuge is used to achieve particle settling, the acceleration due to gravity, g , is no longer valid and must be replaced with centrifugal or radial acceleration, a_R . The radial acceleration is defined as:

$$a_R = w^2 * r = g * G \quad (12)$$

where:

w = rotation speed in radians per second;

r = radius of the centrifuge;

G = unitless term which relates the acceleration due to gravity to the radial acceleration.

The values of G were known for the two different centrifuge speeds used in the fractionation study, and therefore, the radial accelerations were easily calculated.

The values used in Stokes' equation and the calculated equivalent spherical diameter of the smallest particle in the centrate from each fraction are given in Table 12. Terminal velocity of the particles was calculated by dividing the height of the suspending medium by the time the particles were allowed to settle.

The COD in each of the groups listed in Table 11 represents a cumulative COD distribution. For example, the COD content of group II is made up of the COD of group III and the COD fraction between groups II and III. The fraction between groups II and III represent an incremental COD fraction. The incremental COD fractions were calculated for each set of data. With the four groups of cumulative COD given in Table 11, five individual fractions could be calculated. These included Total, Total minus I (Total - I), I minus II (I - II), II minus III (II - III), and the soluble portion as III. Each cumulative group listed in Table 11 was also defined based on the particle size distribution of the centrate after fractionation. Since each group is cumulative, the centrate of each group contains all particles with an equivalent particle diameter less than the smallest particle removed. The cumulative size distribution of particles in each size fraction is given in Table 13.

The incremental size distributions were also determined for each incremental fraction and are given in Table 14. The incremental group I - II contains the largest size range of particles, from 2 to 30 micrometers. However, as will be shown in Chapter V, the I - II group does not always contain the largest percentage of total COD.

Table 12. Summary of values used in calculating the equivalent spherical diameter of the smallest particle removed in each step of fractionation

	Imhoff Cone	Centrifuge @ 1935 G	Centrifuge @ 39100 G
V, m/s	0.0017	0.0015	0.0015
g, m/s ²	9.81		
a, m/s ²		18982	383571
p ₁ ^{a,b} , kg/m ³	1098	1098	1098
p ₂ ^a , kg/m ³	998.2	998.2	998.2
n ^a , kg/m/s ²	0.001002	0.001002	0.001002
d, micrometers	30	2	0.04

^aValues at 20 degrees Celsius.

^bParticles were assumed to have a specific gravity of 1.05.

Table 13. Summary of cumulative particle size fractions

Group	Equivalent Particle Size Distribution
Total	All particles
I	Particles less than 30 micrometers in diameter
II	Particles less than 2 micrometers in diameter
III	Particles less than 0.04 micrometers in diameter

Table 14. Summary of incremental particle size fractions

Group	Equivalent Particle Size Distribution
Total	All particles
Total - I	Particle size diameter > 30 micrometers
I - II	30 micrometers > diameter > 2 micrometers
II - III	2 micrometers > diameter > 0.04 micrometers
III	Particle size diameter < 0.04 micrometers

Gas chromatographic analyses

Gas chromatography was used to analyze the content of the gas produced by each system and the concentration of individual volatile organic acids in feed sludges and reactor contents. Sampling procedures and chromatographic conditions that were used are described in this section.

Gas analysis Gas analysis included the determination of percentages of carbon dioxide, methane, and nitrogen in each system off-gas. The gas chromatographic conditions are listed in Table 15. The microsyringe used in the study was a Model Gastight #1001, Hamilton Co., Reno, Nevada, Pat. No. 3150801, and was equipped with a Hamilton syringe valve.

The general method of gas sampling and analysis was as follows:

1. The syringe valve was opened and the syringe plunger was completely depressed,

Table 15. Operating conditions for gas analysis

Gas chromatograph	Packard Model 7411S
Column	10 ft x 4 mm glass
Packing	Porapak Q, 80/100 mesh
Temperature	40 degrees Celsius
Carrier gas	Helium
Flowrate	25 mL/min
Column head pressure	60 psig
Detector	Thermoconductivity
Temperature	40 degrees Celsius
Bridge current	225 mV
Sensitivity	10 mV
Injector block temperature	80 degrees Celsius

2. The syringe needle was introduced into the gas sampler through the septum,
3. Approximately 0.5 mL of gas was drawn into the syringe,
4. The needle was withdrawn from the gas sampler and the plunger completely depressed,
5. The needle was again introduced into the gas sampler and 1 mL of gas was drawn into the syringe,
6. After approximately 5 seconds, the syringe valve was closed and the needle withdrawn from the gas sampler,
7. The syringe was immediately transferred to the instrument room for analysis,
8. The syringe valve was opened while the plunger was slowly depressed,

9. As the gas volume was reduced to approximately 0.6 mL, the sample was injected into the instrument and analyzed.

Note: The sample for gas analysis could be approximated because percentages of methane and carbon dioxide in the gas sample were measured instead of exact concentrations of each. Theoretically, the percentage of methane and carbon dioxide in various size samples is the same and the size of sample has no bearing on the results.

Volatile Acids Gas chromatography was used to determine concentrations of six volatile organic acids. The operating conditions for the analysis are given in Table 16.

The procedure for analysis of individual volatile acids was as follows:

1. Samples from each reactor were taken as described in Appendix A and subjected to a relative centrifugal force of 39100 G for 30 minutes,
2. Approximately 5 mL of centrate was transferred by pipette to a 7 mL specimen bottle,
3. The centrate in the specimen bottle was acidified to a pH less than or equal to 2 with concentrated sulfuric acid,
4. The specimen bottles were stored at 4 degrees Celsius until the end of the study at which time all the samples were analyzed.

Utilizing the methods described in this chapter, the experimental study produced some very interesting results. Data collected for methane production, concentrations of individual and total volatile organic acids, and particle size distributions were used to compare the two treatment systems. The treatment systems were evaluated at three different HRTs while being fed either homogenized or non-homogenized primary sludge.

Table 16. Operating conditions for individual volatile organic acid analysis

Gas chromatograph	Hewlett Packard Model 5730A
Column	4 ft x 2 mm glass
Packing	GP 60/80 Carbopack C/0.3 % Carbowax 20M/0.1% H ₃ PO ₄
Temperature	120 degrees Celsius
Carrier gas	Nitrogen
Flowrate	50 mL/min
Column head pressure	60 psig
Detector	Flame Ionization
Temperature	200 degrees Celsius
Injector block temperature	200 degrees Celsius
Sample size	1 microliter

CHAPTER V. RESULTS AND DISCUSSION

The experimental study consisted of three phases and included operation of a conventional and a two-phase anaerobic treatment system in parallel at three different hydraulic retention times (HRTs). Each phase was divided into two subphases based on the physical characteristics of the feed sludge. Primary sludge was fed to each system during one subphase and homogenized primary sludge during the other. Data were collected on the performance of each treatment system to determine if particle size reduction improved biological conversion of the feed substrate to methane.

Comparisons were made between the two treatment systems based on methane production, concentrations of individual and total volatile organic acids in each reactor, and the particle size distributions of feed sludges and reactor contents. The chemical oxygen demand (COD) of each particle size fraction was determined for samples of feed sludges and reactor contents at the conclusion of each subphase.

Methane Production

Average methane production rates and standard deviations for both anaerobic systems are given in Table 17. The value of n in Table 17 represents the number of sequential daily gas production rates that were averaged. The gas production rates for phase II were inaccurate due to problems with evaporation of water from the wet test gas meters and therefore, are not included here.

Table 17. Average methane production rates for anaerobic treatment systems during each phase of the experimental study

Phase of Study	Average Methane Production Rate (Std dev, Coeff of Variation, n), L CH ₄ /day	
	Primary Sludge	Homogenized Sludge
Phase I. 15-day HRT		
Conventional System	1.085 (0.100, 9%, 5)	0.639 (0.079, 12%, 5)
Two-phase System	2.540 (0.169, 7%, 5)	2.665 (0.527, 20%, 5)
Phase III. 10-day HRT		
Conventional System	4.663 (0.442, 9%, 5)	4.781 (0.267, 6%, 5)
Two-phase System	5.642 (0.544, 10%, 5)	5.403 (0.450, 8%, 5)

The coefficient of variation was calculated for each average methane production rate and is also given in Table 16. The equation defining the coefficient of variation as a percentage is:

$$\text{Coefficient of Variation} = (s/x) * 100 \quad (13)$$

where

s = standard deviation of the methane production rates;

x = average methane production rate.

Each system was run for at least one hydraulic retention time (HRT) to approach pseudo steady state conditions before calculating an average

methane production rate. A reasonable coefficient of variation of less than or equal to 10 % was desired and achieved in every case except during the homogenized feed study of phase I. However, because the two-phase system had been operating at a 15-day HRT for over 5 hydraulic retention times, it was assumed to be at pseudo steady state.

COD Measurements

The results given in Table 17 are based on the influent and effluent COD concentrations of each system, the methane percentage of the off-gas of each system, and the daily feed volume of each system. The theoretical methane production rates and the methane production rates per gram of chemical oxygen demand (COD) applied for the various phases of the project are summarized in Table 18. A complete table listing all parameters used in calculating methane production rates is given in Appendix C. Methane production values are not shown for the twenty day HRT study due to the measurement problems in determining average daily methane production rates.

The influent and effluent COD concentrations from each system were determined once per week. Influent COD values were highly variable throughout the study and an average value was calculated for each separate phase. The method used in calculating the average influent COD is outlined in Appendix C with the values tabulated in Table C-3. Due to the small reactor volume, relatively short detention time, and the variability of the influent COD concentration, the effluent COD concentration was also variable. Furthermore, highly variable effluent COD concentrations can be directly related to the extremely high

concentrations of volatile organic acids that existed in all reactors during the homogenized feed studies and carried over into the primary sludge feed studies.

Several trends can be seen in the gas production data presented in Table 18. The two-phase system had slightly higher gas production than did the conventional system in every case. In comparing the results of the primary sludge feed study and the homogenized feed study, the performance of both the conventional and two-phase systems decreased when treating the homogenized substrate. This can be attributed to the high volatile acids concentrations which existed in both systems when treating the homogenized substrate. Volatile acids concentrations reached inhibitory levels thereby decreasing treatment efficiency as will be discussed later.

Decreased gas production from the conventional reactor during the homogenized feed subphase of the 15-day HRT study can also be attributed to a bicarbonate alkalinity in the reactor above the normal maximum operating limit of 5000 mg/L as CaCO_3 (see Figure 2, Chapter III). Sodium bicarbonate was added to control low pH resulting from high volatile acids concentrations and toxic bicarbonate levels resulted from the presence of excess sodium bicarbonate.

Volatile Acids Analysis

The concentrations of individual volatile organic acids in feed sludges and reactor contents were analyzed to characterize the effects of particle size reduction and changing HRT on treatment system

Table 18. Values for calculated methane production for each treatment system during the 15-day and 20-day HRT phases of the experimental study

Phase of Study	Theoretical Methane Production ^a , L CH ₄ /d		Methane Production, L CH ₄ /g COD applied	
	Primary Sludge	Homogenized Sludge	Primary Sludge	Homogenized Sludge
Phase I. 15-day HRT				
Conventional System	1.17	0.53	0.11	0.05
Two-phase System	1.56	1.24	0.15	0.12
Phase III. 10-day HRT				
Conventional System	3.38	2.45	0.17	0.15
Two-phase System	4.66	3.06	0.21	0.17

^aCalculated by multiplying actual value for COD destroyed by the theoretical gas production value, 0.35 L CH₄/g COD destroyed.

performance. Samples were taken from each reactor and analyzed for six individual volatile acids. The results of the analyses performed for each reactor and the concentrations of total volatile organic acids concentration in mg/L as acetic acid are given in Appendix E.

In the systems processing homogenized sludge, the concentration of total volatile acids was higher than in the systems processing non-homogenized sludge. The total volatile acids concentration in the reactors during the homogenized feed studies is well above the level that is considered to be inhibitory, 6000 mg/L as acetic acid (23), and

may help explain the inconsistencies in the gas production data. Inhibitory volatile organic acids concentrations existed in the acidogenic reactor, which was operated at a constant HRT of one day throughout the study.

Total volatile organic acids concentrations in the acidogenic reactor ranged from 1841 to 14366 mg/L as acetic acid during the primary sludge feed studies to between 10760 and 38364 mg/L during the homogenized feed studies. Therefore, during the homogenized feed studies, the methanogenic reactor was being fed effluent from the acidogenic reactor which had a total volatile acids concentration in excess of 6000 mg/L.

When comparing the concentrations of individual volatile acids in each reactor, the methanogenic reactor of the two-phase system and the conventional reactor differed markedly. A complete table of the individual volatile organic acids concentrations for each reactor is given in Appendix E. A comparison of the individual volatile acid concentrations for the methanogenic and conventional reactors for each feeding regime of the 20-day HRT study is given in Table 19.

The methanogenic reactor was able to convert the longer chain organic acids such as Iso Butyric, Iso Valeric, and N-Valeric to the shorter chain propionic and useable acetic acid more efficiently than the conventional reactor in both feed studies. However, during the homogenized feed study both systems contained total volatile organic acid concentrations in excess of the inhibitory limit of 6000 mg/L as acetic acid, thereby resulting in decreased treatment efficiency in both

Table 19. Comparison of the individual volatile organic acids concentrations in the conventional and methanogenic reactors during phase II

	Concentration, mg/L as Acetic Acid			
	Primary Sludge (11/29)		Homogenized Sludge (11/13)	
	Conv. Reactor	Meth. Reactor	Conv. Reactor	Meth. Reactor
Acetic Acid	1201	748	13748	10463
Propionic Acid	702	90	574	1758
Iso Butyric Acid	52	ND ^a	47	ND
N-Butyric Acid	2104	22	145	2372
Iso Valeric Acid	91	ND	34	ND
N-Valeric Acid	35	ND	50	16
Total as Acetic Acid	4185	860	14598	14609

^a Not Detected.

systems.

Data from the particle size fractionation studies of phases II and III, 20-day and 10-day HRTs, further explain the transformations that occurred when treating the homogenized substrate as compared to the non-homogenized substrate.

Particle Size Fractionation

The fractionation of feed sludges and reactor contents was performed as described in Chapter IV. Data were collected at the end of each subphase of the study. A cumulative distribution of COD over various particle size fractions was obtained for samples of feed sludges and reactor contents. The incremental COD concentration of each fraction was calculated from the cumulative values. The notation used for the incremental data is given in Table 20. Data collected for the cumulative COD distributions and the calculated incremental fractions COD for each sample are tabulated in Appendix D. Samples were collected only once for each study and therefore, values are assumed to represent trends in the anaerobic systems and not exact transformations of the COD.

The fractionation technique used in the particle size distribution studies involved sedimentation followed by centrifugation. Feed sludges and reactor contents were analyzed for particle size distributions by diluting a sample of each and mixing thoroughly. The solution was then allowed to settle in a standard Imhoff cone with two samples of supernatant taken after 30 minutes. One sample was analyzed for COD and the other centrifuged at two different speeds. Samples of centrate after centrifuging at each speed were also analyzed for COD. The fractionation process resulted in a distribution of three size fractions along with the total COD of each sample. Equivalent spherical diameters of the smallest particle removed in each step of the fractionation were then calculated using Stokes' equation for sedimentation.

Table 20. Summary of incremental particle size fractions

Group	Equivalent Particle Size Distribution
Total	All particles
Total - I	Particle size diameter > 30 micrometers
I - II	30 micrometers > diameter > 2 micrometers
II - III	2 micrometers > diameter > 0.04 micrometers
III	Particle size diameter < 0.04 micrometers

The separations achieved were approximate due to inherent problems existing with centrifugation as initial centrifugation of a sample will remove a percentage of all particle size fractions. However, relatively dilute samples were analyzed throughout the study and most of the large settleable particles were removed by sedimentation, and therefore, the percentage of each fraction removed by the interaction of particles during centrifugation was assumed minimal.

Phase III; Primary Sludge Feed Study

After completion of the 20-day hydraulic retention time (HRT) primary sludge feed study, the 10-day HRT primary sludge feed study was initiated. Throughout the study, the methanogenic reactor of the two-phase system operated at an HRT of 9 days and the acidogenic reactor at a one-day HRT. Therefore, the methanogenic reactor operated at an HRT of one day (10%) less than the conventional reactor.

The HRT of 9 days for the methanogenic reactor is below the minimum SRT of 10 days for anaerobic treatment as shown previously in Figure 3, Chapter III. As the SRT drops below the minimum of 10 days, the percent removal of COD drops off rapidly. However, the decreased SRT of the methanogenic reactor did not affect the percent removal of COD as shown in Table 21. The data shown in Table 21 were used to calculate the gas production rates given in Table 18. Decreased treatment efficiency due to high volatile organic acids concentrations is also evidenced in Table 21 where COD removal percentages during the homogenized feed studies are well below those shown previously in Figure 3.

Table 21. Percent COD removal for each treatment system during the 10-day and 15-day HRT phases of the experimental study

Phase of Study	Percent of COD Removed	
	Primary Sludge	Homogenized Sludge
Phase I. 15-day HRT		
Conventional System	32	15
Two-phase System	42	35
Phase III. 10-day HRT		
Conventional System	49	43
Two-phase System	61	48

Incremental COD analysis The incremental COD fractions for the feed sludge and reactor contents during the primary sludge feed subphase of the 10-day HRT study are shown in Figure 6. For plotting purposes, apparent minimum and maximum particle sizes of 0.001 and 500 micrometers, respectively, were assumed. The percentage of total COD contained in each fraction is also given in Table 22.

In reviewing the results shown in Figure 6 for the methanogenic and the conventional reactors, it can clearly be seen that the methanogenic reactor converted the larger particles to the smaller size ranges more efficiently than the conventional reactor. Comparing percentages of total COD from Table 22, ninety (90) percent of the total COD in the conventional reactor was contained in the greater than 2 micrometer range, whereas the same size range in the methanogenic reactor contained only sixty-nine (69) percent. Consequently, the methanogenic reactor had a greater percentage of COD in the smaller, more useable size ranges. Concentrations of COD in the less than 0.04 micrometer range (assumed soluble COD) in the methanogenic reactor are noticeably higher than in the conventional reactor, nineteen (19) percent versus four (4) percent of the total COD. The increased amount of assumed soluble COD in the methanogenic reactor coincides with the increased methane production rates of the two-phase system over the conventional system shown previously in Table 18.

Phase III; Homogenized Feed Study

Both treatment systems had been operating at a 10-day HRT for 21 days prior to beginning the homogenized feed subphase of the 10-day HRT

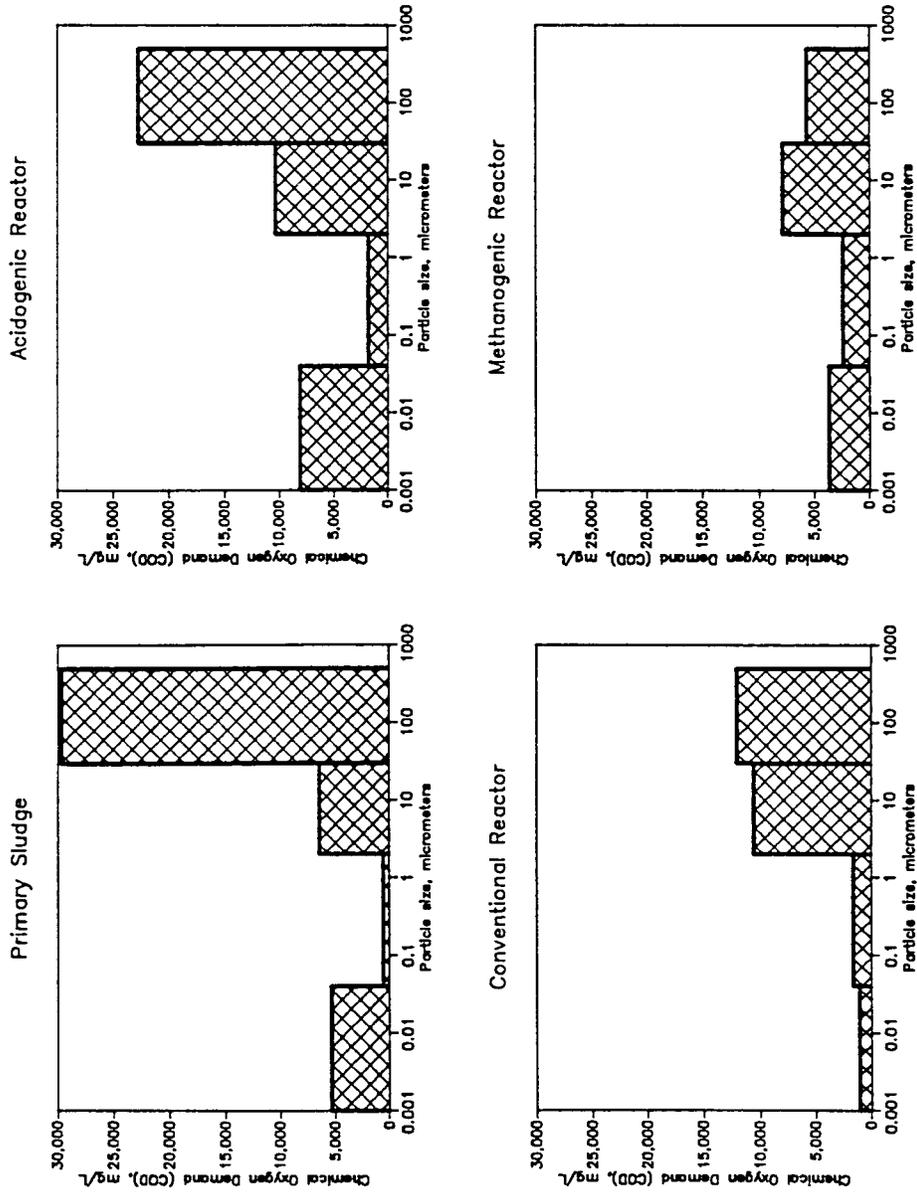


Figure 6. Incremental COD distributions for samples taken during phase III, 10-day HRT, during the primary sludge feed study, with equivalent particle size diameters of each fraction defined in Table 20 of Chapter V. The fraction between 0.001 and 0.04 micrometers represents the more soluble portion of each sample

Table 22. Percent COD contained in incremental fractions of samples from each reactor during the primary sludge feed study of phase III

Group Fraction	Size Range, micrometers	Percent of Total COD, mg/L [?]		
		Feed Sludge	Conventional Reactor	Methanogenic Reactor
Total - I	> 30	71	48	29
I - II	2 to 30	15	42	40
II - III	0.04 to 2	1	6	12
III	< 0.04	13	4	19

study. The systems were operated utilizing the homogenized feed substrate for 11 days. Decreased treatment efficiency in the methanogenic reactor was evidenced during the homogenized feed study and most likely due to the concentration of volatile acids exceeding the inhibitory limit of 6000 mg/L as Acetic Acid.

Incremental COD analysis The incremental COD fractions for the homogenized feed subphase of the 10-day HRT study are shown in Figure 7. A summary of the data for the percent of COD contained in incremental fractions of feed sludges and reactor contents is also given in Table 23.

Immediately it can be seen from Figure 7 that the acidogenic reactor transformed much of the larger particle size material towards

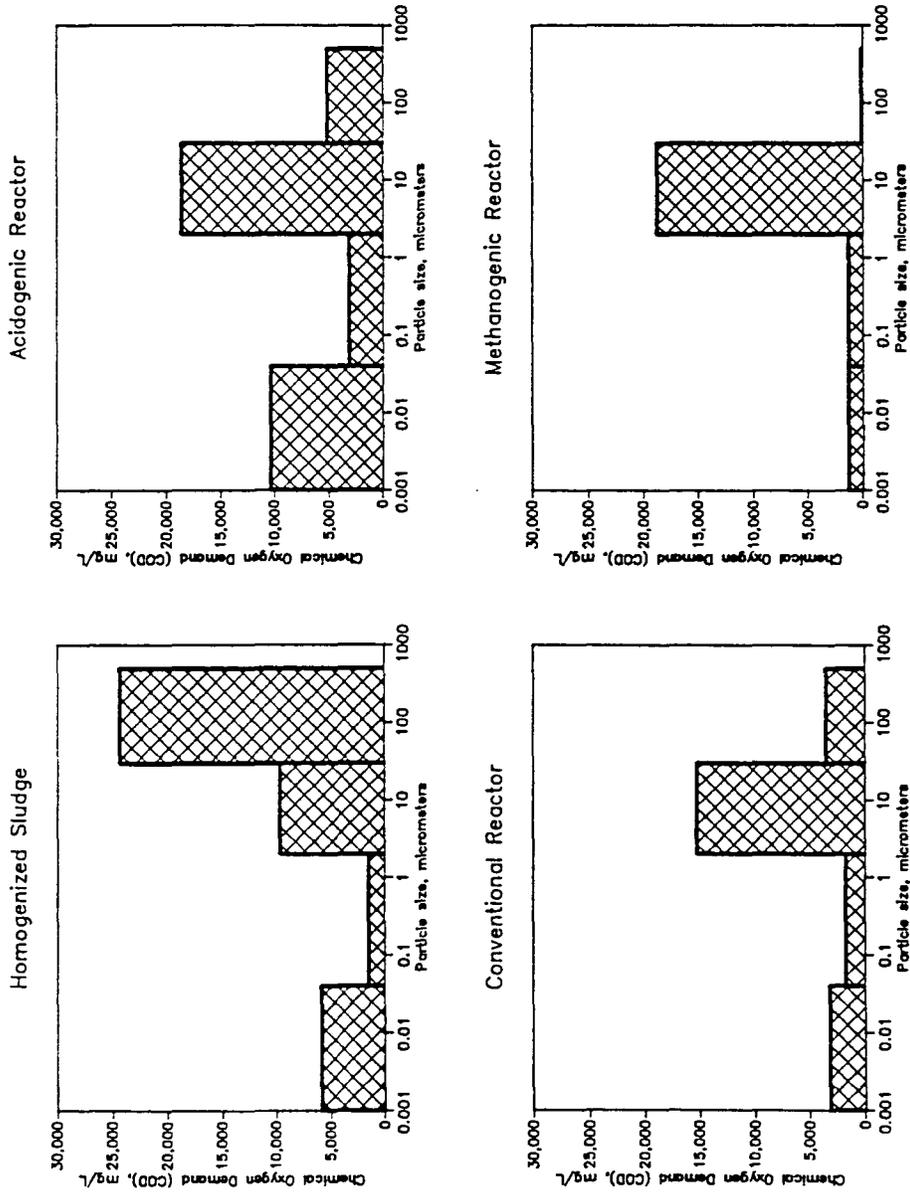


Figure 7. Incremental COD distributions for samples taken during phase III, 10-day HRT, during the homogenized feed study, with equivalent particle size diameters of each fraction defined in Table 20 of Chapter V. The fraction between 0.001 and 0.04 micrometers represents the more soluble portion of each sample

Table 23. Percent COD contained in incremental fractions of samples from each reactor during the homogenized sludge feed study of phase III

Group Fraction	Size Range, micrometers	Percent of Total COD, mg/L		
		Feed Sludge	Conventional Reactor	Methanogenic Reactor
Total - I	> 30	59	15	<1
I - II	2 to 30	23	65	88
II - III	0.04 to 2	4	7	6
III	< 0.04	14	13	6

the smaller size ranges before being fed to the methanogenic system. More importantly, the COD contained in the smallest size fractions in the acidogenic reactor have been reduced in the methanogenic reactor to product methane. However, transformation of COD from the larger size ranges, 2 to 30 micrometers, to the smaller size ranges did not occur. Eighty-eight (88) percent of the total COD in the methanogenic reactor remained in the larger non-useable size ranges. Similar transformations of COD occurred in the conventional reactor when compared to the two-phase system.

Large amounts of COD were converted from the greater than 30 micrometer range to the 2 to 30 micrometer range but further reduction occurred at a much slower rate. The COD contained in the 2 to 30 micrometer range accounted for sixty-five (65) percent of the total COD

in the conventional reactor as stated in Table 23. Incomplete conversion of the substrate to the soluble fraction was likely caused by the high volatile acids concentrations which existed in both treatment systems throughout the homogenized feed subphase.

Phase II; Primary and Homogenized Sludge Feed Studies

The results of the particle size fractionation studies for both feed subphases of phase II, 20-day HRT, are included in Appendix F. Incremental COD distributions are presented and substantiate the results obtained during the 10-day HRT study.

Data collected during the particle size fractionation studies were also used to compare rates of hydrolysis in the acidogenic reactor of the two-phase system. Effects of reducing the particle size of the influent substrate was evaluated for both feeding regimes, homogenized and non-homogenized sludge.

Hydrolysis Rate Constants

As presented previously in Chapter III, the rate of hydrolysis can be defined for any completely mixed continuous flow reactor. For efficient waste treatment to occur, particulate COD material must first be transformed to a soluble substrate which is then converted to methane and carbon dioxide. Hydrolysis of the large particulates by acidogenic bacteria is the first step in the treatment process and considered by some to be the rate-limiting step in the anaerobic degradation process (9,10,28,29). Therefore, increasing the rate of hydrolysis of the particulates may increase the rate at which the waste is converted.

Hydrolysis rate constants were calculated as shown in Appendix G for the acidogenic reactor of the two-phase system. The purpose of the acidogenic reactor is to provide an optimum environment for hydrolysis and subsequent acidification of the waste to occur. This provided an excellent basis for comparing the hydrolysis rates when treating homogenized and non-homogenized primary sludge. As previously stated, the acidogenic reactor was operated at a one-day HRT throughout the study and the calculated hydrolysis rate constants are given in Table 24.

Comparing the results presented in Table 24, a dramatic increase occurred in the rate of hydrolysis during the phase III study. The rate increased from 0.082 days^{-1} to 0.145 days^{-1} when the feed sludge was homogenized. Improved hydrolytic conversion was not as dramatic during the phase II study but the rate of hydrolysis did improve when treating homogenized feed sludge over the non-homogenized substrate.

In this study the effects of particle size reduction on anaerobic digestion were evaluated and two treatment systems were compared: a conventional and a two-phase system. Although decreased treatment efficiency occurred in almost every phase of the study due to high concentrations of volatile organic acids in both treatment systems, some significant conclusions and recommendations can be derived from the results.

Table 24. Hydrolysis rate constants for the acidogenic reactor of the two-phase system during both feed studies of phases II and III

Phase of Study	Hydrolysis Rate Constant, days ⁻¹	
	Primary Sludge	Homogenized Sludge
Phase II. 20-day HRT	0.066	0.083
Phase III. 10-day HRT	0.082	0.145

CHAPTER VI. CONCLUSIONS AND RECOMMENDATIONS

Two anaerobic treatment systems, a conventional and a two-phase system, were compared in this study and the effects of reducing the particle size of the influent feed to each system evaluated. The conclusions of the study are as follows:

1. There is no difference in gas quality from digesters treating primary sludge or homogenized primary sludge,
2. Reduction of the particle size of the influent waste stream causes a shift in the size distribution of the chemical oxygen demand (COD) toward the smaller particle size ranges,
3. Reduction of the particle size of the influent waste stream causes an increase in the volatile acids content of the digesting sludge,
4. The two-phase system can convert larger particles of COD to a more useable substrate more efficiently than the conventional system.

Inhibitory concentrations of volatile acids existed in both treatment systems throughout much of the study. This resulted in decreased treatment efficiency and difficulty in obtaining supporting data to evaluate the effects that particle size reduction had on each of the treatment systems. Therefore, to improve the quality of the data derived from future studies, the following recommendations are made:

1. Collection of feed sludges should be done on a batch basis in order to obtain a more uniform feed COD,
2. A gas mixing device should be used to prevent particle size reduction from taking place due to mechanical mixing,
3. A method of particle size reduction should be used to achieve a much smaller particle size distribution of the influent substrate,

4. Methods of smaller particle size fractionation should be used to obtain values relating to the more soluble portion of each sample.

BIBLIOGRAPHY

1. Balmat, J. L. "Biochemical Oxidation of Various Particulate Fractions of Sewage." Sewage and Industrial Wastes, 29 (7) (1957), 757-761.
2. Bryant, M. P. "Microbial Methane Production - Theoretical Aspects." Journal of Animal Science, 48 (1979), 193-201.
3. Christensen, Douglas R. and Perry L. McCarty. "Multi-Process Biological Treatment Model." Journal of the Water Pollution Control Federation, 47 (11) (1975), 2652-2664.
4. Cohen, A., A. M. Breure, J. G. Van Andel, and A. Van Deursen. "Influence of Phase Separation on the Anaerobic Digestion of Glucose-I Maximum COD-Turnover Rate during Continuous Operation." Water Research, 14 (1979), 1439-1448.
5. Cseh, Tibor, Lajos Czako, Jenő Toth, and Robert P. Tengerdy. "Two-Phase Anaerobic Fermentation of Liquid Swine Waste to Methane." Biotechnology and Bioengineering, 26 (12) (1984), 1425-1429.
6. Dague, Richard R., Ross E. McKinney, and John T. Pfeffer. "Anaerobic Activated Sludge." Journal of the Water Pollution Control Federation, 38 (2) (1966), 220-226.
7. Dague, Richard R., Ross E. McKinney, and John T. Pfeffer. "Solids Retention in Anaerobic Waste Treatment Systems." Journal of the Water Pollution Control Federation, 42 (2) (1970), R29-R46.
8. Daniels, Lacy. "Biological Methanogenesis Physiological and Practical Aspects." Trends in Biotechnology, 2 (1984), 91-98.
9. Eastman, John A. and John F. Ferguson. "Solubilization of Particulate Organic Carbon during the Acid Phase of Anaerobic Digestion." Journal of the Water Pollution Control Federation, 53 (3) (1981), 352-366.
10. Ghosh, Sambhunath, John R. Conrad, and Donald L. Klass. "Anaerobic Acidogenesis of Wastewater Sludge." Journal of the Water Pollution Control Federation, 47 (1) (1975), 30-45.
11. Hindin, Ervin and Gilbert H. Dunstan. "Effect of Detention Time on Anaerobic Digestion." Journal of the Water Pollution Control Federation, 32 (9) (1960), 930-938.

12. Holland, F. A. and F. S. Chapman. Liquid Mixing and Processing in Stirred Tanks. 1st ed. New York: Reinhold Publishing Corporation, 1966.
13. Johnson, Lyle D. "Inhibition of Anaerobic Digestion by Organic Priority Pollutants." Ph.D. dissertation, Iowa State University, 1981.
14. Kleinstreuer, C. and T. Poweigha. "Dynamic Simulator for Anaerobic Digestion Processes." Biotechnology and Bioengineering, 24 (1982), 1941-1951.
15. Lawler, Desmond F., Yoon Jin Chung, Shiaw-Jy Hwang, and Barbara A. Hull. "Anaerobic Digestion: Effects on Particle Size and Dewaterability." Journal of the Water Pollution Control Federation, 58 (2) (1986), 1107-1117.
16. Levine, Audrey D., George Tchobanoglous, and Takashi Asano. "Benefits of Particle Size Management for Biological Wastewater Treatment." Prepared for Presentation at the 1985 A.S.C.E. National Conference on Environmental Engineering, Boston, Massachusetts, July, 1985.
17. Levine, Audrey D., George Tchobanoglous, and Takashi Asano. "Characterization of the Size Distribution of Contaminants in Wastewater: Treatment and Reuse Implications." Journal of the Water Pollution Control Federation, 57 (7) (1985), 805-816.
18. McCarty, Perry L. and Daniel P. Smith. "Anaerobic Wastewater Treatment." Environmental Science Technology, 4th ser., 6 (1986), 1200-1206.
19. McCarty, Perry L. "Anaerobic Waste Treatment Fundamentals." Public Works, 1st ser., 4 (1964), 107-112.
20. McCarty, Perry L. "Anaerobic Waste Treatment Fundamentals." Public Works, 2nd ser., 4 (1964), 123-126.
21. Morgan, Philip F. "Studies of Accelerated Digestion of Sewage Sludge." Journal of the Water Pollution Control Federation, 26 (4) (1954), 462-478.
22. Murk, John S., Jerry L. Frieling, Liberato D. Tortorici, and Clyde C. Dietrich. "Use of Bench-Scale Digesters to Evaluate Full-Scale Digester Performance." Journal of the Water Pollution Control Federation, 52 (11) (1980), 2709-2716.

23. Parkin, Gene F. and William F. Owen. "Fundamentals of Anaerobic Digestion of Wastewater Sludges." Journal of Environmental Engineering Division, American Society of Civil Engineers, 112 (EE1) (February 1986), 867-920.
24. Pierce, William H. "Effects of Particle Size on the Anaerobic Decomposition of Volatile Solids." Master of Science Thesis, Iowa State University, 1961.
25. Sawyer, C. N. and H. E. Schmidt. "High-Rate Sludge Digestion." Journal of the Boston Society of Civil Engineers, 42 (1) (1955), 1-17.
26. Sawyer, C. N. and H. K. Roy. "A Laboratory Evaluation of High-Rate Sludge Digestion." Journal of the Water Pollution Control Federation, 27 (12) (1955), 1356-1363.
27. Schlenz, Harry E. "Discussion: Loading to Failure of a Pilot High-Rate Digester." Journal of the Water Pollution Control Federation, 27 (2) (1955), 133-142.
28. Speece, Richard E. "Anaerobic Biotechnology for Industrial Wastewater Treatment." Environmental Science Technology, 17 (1983), 416A-427A.
29. Speece, Richard E. "Environmental Requirements for Anaerobic Digestion." Prepared for presentation at the Association of Environmental Engineering Professors Workshop on Anaerobic Treatment Processes, Purdue University, May, 1985.
30. Standard Methods for the Examination of Water and Wastewater. 16th ed. Washington, D. C.: American Public Health Association, 1985.
31. Steele, E. W. and Terence J. McGhee. Water Supply and Sewerage. New York: McGraw-Hill, Inc., 1979.
32. Switzenbaum, Michael S. "Anaerobic Fixed Film Wastewater Treatment." Enzyme Microbiology Technology, 5 (7) (1983), 242-250.
33. Tchobanoglous, George. Wastewater Engineering: Treatment, Disposal, Reuse. 2nd ed. New York: McGraw-Hill, Inc., 1979.
34. Torpey, Wilbur N. "High-Rate Digestion of Concentrated Primary and Activated Sludge." Journal of the Water Pollution Control Federation, 26 (4) (1954), 479-494.

35. Torpey, Wilbur N. "Loading to Failure of a Pilot High-Rate Digester." Journal of the Water Pollution Control Federation, 27 (2) (1955), 121-133.
36. Viessman, Warren, Jr. Water Supply and Pollution Control. 2nd ed. New York: Harper and Row, 1985.

APPENDIX A.

DAILY REACTOR FEEDING PROCEDURES

Three reactors were used in this research project. The reactor numbers, types, and feeding schedules are summarized in Table A-1.

Table A-1. Summary of reactors feeding schedule

Reactor Number	Type	Feeding Schedule	Procedure Description Appendices
1	Acidogenic	every 8 h	A-1;A-3;A-5
2	Methanogenic	every 24 h	A-1;A-4
3	Conventional	every 24 h	A-1;A-2

Reactor 1, the acidogenic reactor, was operated with a 1 day detention time throughout the study. Therefore, reactor 1 was fed three times a day at approximate 8 hour intervals. Feeding of reactors 2 and 3, the methanogenic and conventional reactors, respectively, was accomplished on a batch basis (once every 24 hours). The feeding took place in the early morning to allow for analysis of reactor contents the remainder of the day. The morning feeding for reactor 1 differed from that of the afternoon and evening feedings in that the effluent from reactor 1 was fed to reactor 2 only in the morning and completely wasted at the other times.

EQUIPMENT USED:

1. Three 5 L plexiglas anaerobic reactors as shown in Figure 4-1
2. Three Talboy model variable speed electric mixer motors with 3.5 cm diameter three blade marine type propellers attached
3. Two 500 mL bottles fitted with stoppers and glass tubing as shown in Figure 4-3 and filled half way with water acidified to pH 1 with sulfuric acid
4. Variable speed Masterflex pump Model no. 7553-30
5. Two stainless steel size 18 Masterflex pump heads Model no. 7018-21
6. Tygon food Masterflex tubing
7. Two glass "T"'s
8. Low gas permeable tygon tubing
9. One 200 mL glass feed funnel
10. One 2 L waste beaker
11. Four 500 mL waste beakers
12. Eight plastic Erlenmeyer flasks with stoppers
13. Four 50 mL glass beakers

Procedure

The daily reactor feeding procedures are detailed in Appendices A-1, A-2, A-3, A-4, and A-5.

Appendix A-1.

Daily Data Recording and Sampling

Daily data collection for process control of each reactor included recording of date and time of data collection. Wet Test meter readings were recorded each day along with temperature and pH measurements. Digested sludge samples from each reactor and a feed sample were taken periodically and analyzed for COD, alkalinity, and Volatile Acids. Also, samples of the off-gas were analyzed for gas composition at regular intervals. A list of the parameters measured and the frequency of measurement is given in Table A-2.

Table A-2. Summary of sampling parameters and frequency of measurement

Parameter	Frequency of Measurement
pH	every day
Temperature	every day
COD	beginning and end of each run, and every 7 days in between
Alkalinity	same frequency as COD
Volatile Acids	same frequency as COD
Gas Composition	every 7 days

The feeding apparatus used and the feed and waste valves of a reactor are shown in Figure A-1.

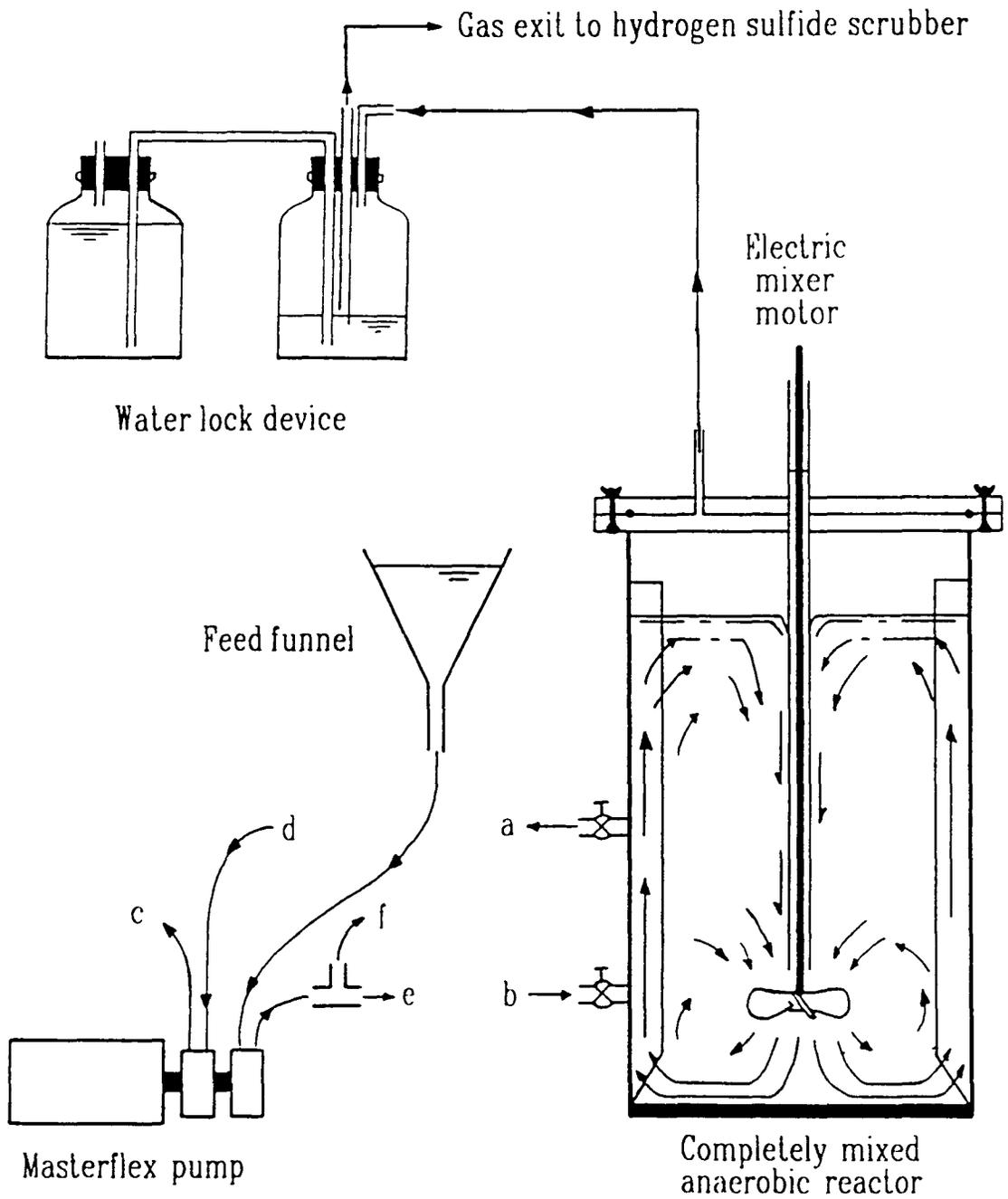


Figure A-1. Feeding apparatus including a) reactor waste valve, b) reactor feed valve, c) pump waste tube outlet, d) pump waste tube inlet, e) pump feed tube inlet, and f) pump feed tube waste line.

Procedure

1. Record day, date, and time.
2. Record temperature of the room.
3. Record the readings of each of the Wet Test meters.
4. Open valve 'a' and waste 10 mL of digested sludge from each reactor.
5. Obtain a sample from each reactor for pH and temperature measurement by opening valve 'a' to release 20 mL of digested sludge into a 50 mL beaker.
6. Measure the temperature of each sample immediately and record.
7. After collecting all three samples, collect a 20 mL feed sample and run pH determinations on each, recording as analyzed.
8. If alkalinity measurements are to be taken, obtain a sample from each reactor by opening valve 'a' to release 50 mL of digested sludge into a Erlenmeyer flask and stopper immediately.
9. If COD measurements are to be taken, obtain a sample from each reactor by opening valve 'a' to release approximately 150 mL of digested sludge into a beaker.
10. If Volatile Acid measurements are to be taken, obtain a sample as in step 7.
11. Refer to Appendix A-2 for the procedure outlining the feeding of the conventional reactor 3.

Appendix A-2.

Daily Feeding Procedure for the Conventional
System (Reactor 3)

After the daily recording of data and sampling in Appendix A-1 were completed, the conventional reactor (reactor 3) was fed its daily feed volume of sludge. The procedure for feeding reactor 3 refers to the feeding apparatus and reactor configuration as shown in Figure A-1.

Procedure

12. Connect point 'd' to valve 'a' on reactor 3.
13. Turn off mixer.
14. Open valve 'a' and turn pump on.
15. Set pump speed at 10 rpm to deliver approximately 40 mL per minute.
16. Allow digested sludge to be withdrawn into a beaker until the level in the reactor reaches the mark corresponding to the given detention time and subsequent volume of waste given in Table A-3.
17. Turn the pump off and close valve 'a'.
18. Disconnect 'd' and 'a' and connect 'e' and 'b'.
19. Fill feed funnel with sludge and open pinch clamp on waste tube 'f'.
20. Turn the mixer on to the 20% setting of a maximum 7500 rpm.

Note: It is important to prevent air from entering the reactor and steps 17 through 25 must be followed carefully to prevent reactor upset.

Table A-3. Distance conventional reactor level drops in relation to various volumes of sludge withdrawn at specified detention times

Detention Time, d	Volume of Sludge Withdrawn, mL	Distance reactor level drops, cm
10	400	2.19
15	266	1.46
20	200	1.1

21. Open valve 'b' allowing digested sludge to fill the line and begin wasting out of tube 'f' and into the waste beaker.
22. Close valve 'b' and turn pump on at the 10 rpm setting.
23. Allow feed sludge to waste out of tube 'f' until no air bubbles can be seen trapped in the clear feed line.
24. Turn off pump and close pinch clamp on waste tube 'f'.

Note: The waste tube 'f' must remain approximately vertical out of the feed line in order to allow any air trapped in the system to rise up into the tube and be replaced by the sludge present in the tube.

25. Open valve 'b' and turn pump on in reverse to pull out any air which may have been caught in the valve.
26. Turn pump off and return the switch to forward.
27. Turn the pump on and begin feeding the reactor.

Note: Watch feed line to make sure the line does not clog. If this should happen, carefully switching the pump between

forward and reverse modes, making sure to pause between modes, should unclog the line. The pump speed can also be increased to help unclog the feed line.

28. Pay close attention to the feed funnel and fill with feed sludge as the level drops.
29. As the level in the reactor approaches the 4 L mark, the mixer is turned off in order to see the level more clearly.
30. The level in the reactor can be adjusted to exactly 4 L by using the variable speed pump controller.
31. Upon reaching the 4 L mark, turn the pump off and close the valve at 'b'.
32. Disconnect points 'e' and 'b' and turn pump on to waste the remaining feed sludge out of the feed line and back into the feed jug.
33. Turn the mixer on to the 20% setting.
34. Refer to Appendix A-3 for the feeding procedure for the two-phase system.

Appendix A-3.

Morning Feeding Procedure for the Two-phase System
Acidogenic Reactor (Reactor 1)

After the daily recording of data and sampling in Appendix A-1 were completed, the two-phase system acidogenic reactor (reactor 1) was fed its morning volume of sludge. The procedure for the morning feeding of reactor 1 refers to the feeding apparatus and reactor as shown in Figure A-1. Appendix A-5 contains the procedure for the daily afternoon and evening feeding of reactor 1.

Procedure

Note: Steps 12 through 34 refer to Appendix A-2.

35. Connect point 'd' to valve 'a' on reactor 1.
36. Turn off mixer.
37. Open valve 'a' and turn pump on.
38. Set pump at 10 rpm to deliver approximately 40 mL per minute.
39. Allow digested sludge to be withdrawn into a beaker up to an amount in excess of that which has to be fed into reactor 2. The volume of effluent from reactor 1 which must be fed to reactor 2 is given in Table A-4 along with approximate quantities withdrawn to ensure adequate volume for priming of the feed lines.

Note: The amount wasted from reactor 1 must be tallied by summing the volumes withdrawn in steps 3,4,7 through 9, and 39.

This amount is then subtracted from 1330 mL to determine the amount of additional sludge to be wasted from reactor 1.

Table A-4. Approximate volume of sludge to be withdrawn from reactor 1 for feed into reactor 2 at specified detention time

Detention Time, d	Volume fed to reactor 2, mL	Approximate Volume withdrawn from reactor 1, mL
9	444	540
15	266	360
19	210	310

40. Turn the pump off and close valve 'a'.
41. Turn the mixer on to the 20 % setting.
42. Disconnect 'd' and 'a' and connect 'e' and 'b'.
43. Fill feed funnel with sludge and open pinch clamp on waste tube 'f'.
44. Feed as in steps 20 through 28.

Note: Feeding of reactor 1 will continue until the mixed level in the reactor is approximately 2 cm below the 4 L mark. At that time, simultaneous feeding and wasting will be continued until the amount wasted into a beaker equals the amount calculated in the note following step 39.

45. Turn the pump off when the level in the reactor is approximately 2 cm below the 4 L mark.
46. Reconnect point 'd' to valve 'a'.
47. Open valve 'a' and turn pump on at the 10 rpm setting.

48. Waste from the reactor into a beaker the amount calculated in the note following step 39.

Note: The pump will waste faster than it feeds due to the consistency of the treated sludge and therefore, there is no danger of overfeeding the reactor.

49. Turn the pump off and close valve 'a'.

50. Disconnect points 'd' and 'a' and turn pump on to finish feeding by following steps 29 through 33.

51. Refer to Appendix A-4 for the feeding procedure for the two-phase system methanogenic reactor.

Appendix A-4.

Daily Feeding Procedure for the Two-phase System
Methanogenic Reactor (Reactor 2)

After the daily recording of data and sampling in Appendix A-1 and the morning feeding of the two-phase system acidogenic reactor (reactor 1) in Appendix A-3 was completed, the two-phase system methanogenic reactor (reactor 2) was fed. Reactor 1 was fed prior to reactor 2 due to the fact that the effluent of reactor 1 was the feed for reactor 2. The procedure for feeding reactor 2 refers to the feeding apparatus and reactor configuration as shown in Figure A-1.

Procedure

Note: Steps 12 through 34, and 35 through 51 refer to Appendices A-2 and A-3, respectively.

52. Repeat steps 12 through 15.
53. Allow digested sludge to be withdrawn into a beaker until the level in the reactor reaches the mark corresponding to the given detention time and subsequent volume of waste given in Table A-5.
54. Feed as in steps 17 through 33 remembering that the feed sludge for reactor 2 is the effluent collected from reactor 1.
55. Refer to Appendix A-5 for the afternoon and evening feeding procedure for the two-phase system acidogenic reactor (reactor 1).

Table A-5. Distance methanogenic reactor level drops in relation to various volumes of sludge withdrawn at specified detention times

Detention Time, d	Volume of Sludge Withdrawn, mL	Distance reactor level drops, cm
9	444	2.43
15	266	1.46
19	210	1.15

Appendix A-5.

Afternoon and Evening Feeding Procedure for the Two-phase
System Acidogenic Reactor (Reactor 1)

The afternoon and evening feeding procedures was similar to the morning feeding procedure for the two-phase system acidogenic reactor (reactor 1), as described in Appendix A-3. The only difference being that all of the effluent from reactor 1 could be wasted since none had to be fed into reactor 2 in the afternoon or evening. The procedure for the afternoon and evening feeding of reactor 1 refers to the feeding apparatus and reactor configuration as shown in Figure A-1.

Procedure

Note: Steps 12 through 34, 35 through 51, and 52 through 55 refer to Appendices A-2, A-3, and A-4, respectively.

56. Connect point 'e' to valve 'b' on reactor 1.

57. Fill feed funnel with sludge and open pinch clamp on waste tube 'f'.

Note: It is important to prevent air from entering the reactor and step 58 must be followed carefully to prevent reactor upset.

58. Prime feed line by following steps 21 through 26.

59. Connect point 'd' to valve 'a'.

Note: The amount wasted from reactor 1 must be tallied by summing the volumes withdrawn in steps 3, 4, and 7 through 9. This amount is then subtracted from 1330 mL to determine the amount of additional sludge to be wasted from reactor 1.

60. Open valve 'a'.

61. Begin feeding by following steps 27 and 28.

Note: The above procedure is allowing simultaneous feeding and wasting of reactor 1. Wasting will be continued until the amount wasted into a beaker equals the amount calculated in the note following step 59. However, since the pump will waste faster than it feeds, as explained in the note following step 48, an excessive drop in the level of the reactor should be corrected by discontinuing wasting until the level in the reactor is approximately 2 cm below the 4 L mark. If this action is taken, the next step is step 62, if it is not, skip directly to step 63.

62. Resume simultaneous feeding and wasting by following steps 45 through 49.

63. Finish feeding by following step 50.

APPENDIX B.

DATA COLLECTED DURING THE EXPERIMENTAL STUDY

Data collected during each phase of the study were entered into separate spread sheets for easy reference and calculation of the daily gas production rates. As the basic parameters were measured for each reactor, the data were entered into the spread sheet. The daily format allowed for easily referenced data and comparison between sets of data.

Calculation of daily gas production rates included correction of the volume of gas produced to standard temperature and pressure (STP). The equation used was as follows:

$$V = \frac{V_1 * P_1 * T_2}{P_2 * T_1}$$

where

V_2 = Corrected volume of gas produced

V_1 = Uncorrected volume of gas produced

P_1 = Adjusted pressure = atmospheric pressure -
water vapor pressure, mm Hg

P_2 = Standard pressure = 760 mm Hg

T_2 = Standard temperature = 0 degrees Celsius =
273.18 degrees Kelvin

T_1 = Temperature of gas produced, degrees Kelvin

Laboratory data collected during the study are arranged chronologically in Appendices B-1 thru B-6 as summarized in Table B-1.

Table B-1. Location of data collected during each phase of the study

	Phase of Study	Appendix
Phase I.	15-day HRT: Primary sludge feed study	B-1
Phase I.	15-day HRT: Homogenized feed study	B-2
Phase II.	20-day HRT: Homogenized feed study	B-3
Phase II.	20-day HRT: Primary sludge feed study	B-4
Phase III.	10-day HRT: Primary sludge feed study	B-5
Phase III.	10-day HRT: Homogenized feed study	B-6

Individual anaerobic reactors were referenced by number in the laboratory data. The two-phase system consisted of reactors #1 and #2, the acidogenic and methanogenic reactors, respectively. Reactor #3 denotes the conventional reactor used in the study.

Appendix B-1.

Daily Operating Data during Primary Sludge Feed Study of Phase I

Run 1: Raw Primary Sludge; 15-day detention time; 16-day run

1/1R

Date	Time		Interval Time, days	Cumulative Time, days	Temperature, degrees Celsius				Barometric Pressure, mm Hg
	hour	minutes			Room	Reactor #1	Reactor #2	Reactor #3	
07-Sep	9	59			34.8	38.5	36	35.5	742.1
08-Sep	7	47	0.91	0.91	34.8	36	35	34.2	743.6
09-Sep	7	59	1.01	1.92	34.7	35.6	35	35	738.9
10-Sep	7	13	0.97	2.88	34.6	36	36	36	731.5
11-Sep	9	5	1.08	3.96	34.8	35	35.1	35.5	731.4
12-Sep	7	55	0.95	4.91	35	36	36	35	741.7
13-Sep	9	35	1.07	5.98	34.7	35.5	35	35	744.4
14-Sep	10	6	1.02	7.00	34.7	35.8	35	34.7	739.8
15-Sep	9	50	0.99	7.99	35	36.2	35.5	35.3	741.6
16-Sep	8	45	0.95	8.95	35	35.5	35	35.2	747.2
17-Sep	7	15	0.94	9.89	34.5	36.5	36	35.8	740.1
18-Sep	10	43	1.14	11.03	34.5	36.2	35.8	35.5	740.9
19-Sep	7	13	0.85	11.88	34.8	35.5	35.6	34.5	737.6
20-Sep	7	30	1.01	12.90	34.5	35.5	35	35	740.0
21-Sep	8	30	1.04	13.94	34.75	36.5	36	35.5	739.0
22-Sep	9	55	1.06	15.00	35	36.5	36	36	738.1

Run 1: Raw Primary Sludge

2/1R

Date	pH				Wet-Test Meter Readings		Gas Production	
	Feed	Reactor #1	Reactor #2	Reactor #3	Two-phase system, L	Conventional System, cf	Two-phase system, L	Conventional System, cf
07-Sep		5.25	7	6.68	144.523	63.9562		
08-Sep		5.4	7.01	6.7	147.218	64.809	2.695	0.0528
09-Sep		5.4	7.07	6.75	149.29	64.0415	2.072	0.0325
10-Sep		5.2	7.02	6.86	152.068	64.077	2.778	0.0355
11-Sep		5.3	7.12	6.96	154.95	64.0898	2.882	0.0128
12-Sep		5.45	7.1	7.03	157.686	64.1082	2.736	0.0184
13-Sep		5.3	7.07	6.98	160.862	64.1346	2.376	0.0264
14-Sep	5.48	5.2	7.1	7.07	162.222	64.1568	2.16	0.0222
15-Sep	5.35	5.31	7.1	7	164.222	64.1818	2	0.025
16-Sep		5.4	7.2	7.12	166.175	64.1982	1.953	0.0164
17-Sep		5.24	7.16	7.05	169.01	64.2362	2.835	0.038
18-Sep	5	5.3	7.08	6.94	172.165	64.2833	3.155	0.0471
19-Sep		5.1	6.97	6.87	174.5	64.3204	2.335	0.0371
20-Sep		5.05	7.02	6.94	177.799	64.3657	3.299	0.0453
21-Sep		5.4	7.09	6.89	180.849	64.4199	3.05	0.0542
22-Sep	5.92	5.4	7.18	6.97	183.33	64.449	2.481	0.0291

Run 1: Raw Primary Sludge

3/1R

Date	Gas Production @ STP		Interval Time, days	Gas Production Rate @ STP		
	Two-phase System, l	Conventional System, l		Two-phase System, L/d	Conventional System, L/d	
07-Sep						
08-Sep	2.339	1.298	0.91	2.575	1.429	
09-Sep	1.787	0.794	1.01	1.773	0.787	
10-Sep	2.373	0.859	0.97	2.452	0.887	
11-Sep	2.460	0.309	1.08	2.283	0.287	
12-Sep	2.367	0.451	0.95	2.488	0.474	
13-Sep	2.065	0.650	1.07	1.931	0.608	
14-Sep	1.866	0.543	1.02	1.826	0.532	
15-Sep	1.730	0.612	0.99	1.749	0.619	
16-Sep	1.702	0.405	0.95	1.783	0.424	
17-Sep	2.451	0.930	0.94	2.615	0.993	
18-Sep	2.731	1.155	1.14	2.386	1.009	Average Gas Production Rate @ STP
19-Sep	2.010	0.904	0.85	2.353	1.059	-----
20-Sep	2.852	1.109	1.01	2.819	1.096	Two-phase System, L/d
21-Sep	2.631	1.324	1.04	2.526	1.271	Conventional System, L/d
22-Sep	2.136	0.789	1.06	2.017	0.670	-----
						2.540
						1.085

						Standard Deviation

						0.169
						0.108

Run 1: Raw Primary Sludge

6/1R

Date	Volatile Acids, mg/L as Acetic Acid			Digester Gas Analysis % CH ₄ , %CO ₂ , %N ₂ in off-gas	
	Reactor #1	Reactor #2	Reactor #3	Reactors #1 and #2	Reactor #3
07-Sep					
08-Sep					
09-Sep					
10-Sep					
11-Sep				40.26,55.82,3.92	21.22,68.48,4.3
12-Sep					
13-Sep					
14-Sep					
15-Sep				40.83,56.70,2.47	29.9,66.77,3.33
16-Sep					
17-Sep					
18-Sep					
19-Sep					
20-Sep					
21-Sep					
22-Sep					

Run 1: Raw Primary Sludge

7/1R

Date	Comments
07-Sep	Fed 4g Sodium Bicarb /266mL feed to Reactor 2; 6g/266 to Reactor 3
08-Sep	Began feeding new sludge @ 11:00 a.m. Picked up 42 gallons raw primary.
09-Sep	Fed 4g Sodium Bicarb /266mL feed to Reactor 2; 6g/266 feed to Reactor 3
09-Sep	Fed 3.5g Sodium Bicarb /266mL feed to Reactor 2; 8g/266 to Reactor 3
10-Sep	Fed 3.5g Sodium Bicarb /266mL feed to Reactor 2; 8g/266 to Reactor 3
11-Sep	Fed 2g Sodium Bicarb /266mL feed to Reactor 2; 4g/266 to Reactor 3
12-Sep	Fed 2g Sodium Bicarb /266mL feed to Reactor 2; 3.5g/266 to Reactor 3
13-Sep	Fed 2g Sodium Bicarb /266mL feed to Reactor 2; 4.5g/266 to Reactor 3
14-Sep	Fed 1.75g Sodium Bicarb /266mL feed to Reactor 2; 4.25g/266 to Reactor 3
15-Sep	Fed 1.25g Sodium Bicarb /266mL feed to Reactor 2; 4.25g/266 to Reactor 3 Reactor 1 feed air when walked out of room
16-Sep	Fed 2g Sodium Bicarb /266mL feed to Reactor 3
17-Sep	Reactor 3 mixer was off all night
18-Sep	Fed 4g Sodium Bicarb /266mL feed to Reactor 3
19-Sep	Fed 2g Sodium Bicarb /266mL feed to Reactor 2; 4g/266 to Reactor 3
20-Sep	Fed 4g Sodium Bicarb /266mL feed to Reactor 2; 8g/266 to Reactor 3
21-Sep	Fed 4g Sodium Bicarb /266mL feed to Reactor 2; 8g/266 to Reactor 3
22-Sep	Fed 6g Sodium Bicarb /266mL feed to Reactor 3 Began feeding blended sludge; blended 3 cups sludge on high for 10 minutes

Appendix B-2.

Daily Operating Data during Homogenized Feed Study of Phase I

Run 1: Blended Primary Sludge; 15-day detention time; 24-day run; total phase 40 days

1/18

Date	Time		Interval Time, days	Cumulative Time, days	Temperature, degrees Celsius				Barometric Pressure, mm Hg
	hour	minutes			Room	Reactor #1	Reactor #2	Reactor #3	
23-Sep	11	30			34.5	36	35.8	36	736.2
24-Sep	9	57	0.94	0.94	34.5	36	35.5	35.7	731.4
25-Sep	7	25	0.89	1.83	34.5	35.8	35.8	35.3	732
26-Sep	16	0	1.36	3.19	34.4	36	36	35	734.0
27-Sep	9	30	0.73	3.92	35	37	36.5	37	737.0
28-Sep	10	0	1.02	4.94	35.5	38	37	37	734.0
29-Sep	8	15	0.93	5.86	35	36.5	36.5	36.5	735.9
30-Sep	7	30	0.97	6.83	34.5	36.5	35.5	35.2	739.2
01-Oct	8	49	1.05	7.89	34.5	36.5	36	35.8	737.7
02-Oct	9	45	1.04	8.93	35	36	35.8	35.8	735.5
03-Oct	10	0	1.01	9.94	34.5	36	35.8	35.8	735.6
04-Oct	8	15	0.93	10.86	34	36.5	36	35.7	738.3
05-Oct	9	9	1.04	11.90	34.5	35.7	35.7	35.7	739.1
06-Oct	7	38	0.94	12.84	35	35.5	36	35.5	742.0
07-Oct	8	26	1.03	13.87	34.5	35.8	35.7	35.5	734.9
08-Oct	7	20	0.95	14.83	34.6	35	35.6	35.4	738.2
09-Oct	8	52	1.06	15.89	34.5	38	35	35.2	748.3
10-Oct	8	50	1.00	16.89	34.5	36.2	35.5	35.5	744.8
11-Oct	10	30	1.07	17.96	33.5	35	35	34.5	738.7
12-Oct	10	0	0.98	18.94	33.5	38	35.5	34.5	738.6
13-Oct	8	51	0.95	19.89	34.5	35.5	35.2	35.2	740.7
14-Oct	11	11	1.10	20.99	34.5	35	35.5	35.5	739.0
15-Oct	9	19	0.92	21.91	34.8	35.8	36	35.5	744.1
16-Oct	9	35	1.01	22.92	35	38	35	35.2	740.0

Run 1: Blended Primary Sludge

2/18

Date	pH				Net-Test Meter Readings		Gas Production	
	Feed	Reactor #1	Reactor #2	Reactor #3	Two-phase system, L	Conventional System, cf	Two-phase system, L	Conventional System, cf
23-Sep	6.1	5.57	7.1	7.02	185.975	64.476		
24-Sep	6.01	5.52	7.07	7.02	188.635	64.489	2.66	0.013
25-Sep	6.37	5.16	6.95	7	191.268	64.5049	2.633	0.0159
26-Sep		5.5	7.08	7.11	195.398	64.5443	4.13	0.0394
27-Sep	5.8	5.45	7	7	197.916	64.5686	2.518	0.0243
28-Sep	5.8	5.4	7.03	7.09	201.898	64.6116	3.982	0.043
29-Sep	5.9	5.24	7.08	7.08	205.283	64.6932	3.385	0.0816
30-Sep	5.91	5.25	7.1	7.2	208.14	64.7728	2.857	0.0796
01-Oct	5.9	5.3	7.15	7.13	210.015	64.8355	1.875	0.0627
02-Oct	6	5.3	7.01	7.07	213.917	64.862	3.902	0.0265
03-Oct	6.02	5.4	7	7.02	216.646	64.89	2.729	0.028
04-Oct	5.82	5.39	6.9	7	219.35	64.93	2.704	0.04
05-Oct	5.6	5.41	6.9	7	222.181	64.993	2.831	0.063
06-Oct	5.39	5.38	7.02	7	224.851	65.0184	2.67	0.0254
07-Oct	5.94	5.33	7	7.05	227.38	65.043	2.529	0.0246
08-Oct	5.9	5.5	7.01	7.06	229.7	65.0585	2.32	0.0155
09-Oct	5.91	5.5	6.96	7.1	232.788	65.0697	3.088	0.0112
10-Oct	5.96	5.55	7.1	7.1	235.12	65.0852	2.332	0.0155
11-Oct	5.8	5.68	6.97	7.09	240.162	65.1052	5.042	0.02
12-Oct	5.88	5.76	7.03	7.01	244.178	65.1249	4.016	0.0197
13-Oct	5.98	5.74	7	7.01	246.335	65.152	2.157	0.0271
14-Oct	5.9	5.61	7	7.02	249.965	65.1837	3.63	0.0317
15-Oct	5.7	5.57	7	7.03	252.62	65.207	2.655	0.0233
16-Oct	5.91	5.5	6.96	7.1	255.5	65.235	2.88	0.028

Run 1: Blended Primary Sludge

3/18

Date	Gas Production @ STP		Interval Time, days	Gas Production Rate @ STP	
	Two-phase System, L	Conventional System, L		Two-phase System, L/d	Conventional System, L/d
23-Sep					
24-Sep	2.273	0.315	0.94	2.430	0.336
25-Sep	2.252	0.305	0.89	2.517	0.431
26-Sep	3.543	0.957	1.36	2.609	0.705
27-Sep	2.164	0.592	0.73	2.968	0.811
28-Sep	3.483	1.041	1.02	3.334	1.020
29-Sep	2.985	1.984	0.93	3.134	2.140
30-Sep	2.467	1.947	0.97	2.547	2.010
01-Oct	1.616	1.530	1.05	1.532	1.451
02-Oct	3.347	0.644	1.04	3.222	0.620
03-Oct	2.345	0.681	1.01	2.321	0.674
04-Oct	2.336	0.979	0.93	2.520	1.056
05-Oct	2.444	1.541	1.04	2.356	1.485
06-Oct	2.311	0.623	0.94	2.467	0.665
07-Oct	2.171	0.598	1.03	2.101	0.579
08-Oct	2.000	0.370	0.95	2.096	0.397
09-Oct	2.700	0.277	1.06	2.537	0.261
10-Oct	2.029	0.382	1.00	2.032	0.382
11-Oct	4.365	0.490	1.07	4.082	0.459
12-Oct	3.477	0.483	0.98	3.551	0.493
13-Oct	1.867	0.664	0.95	1.960	0.698
14-Oct	3.134	0.775	1.10	2.856	0.706
15-Oct	2.306	0.573	0.92	2.500	0.621
16-Oct	2.486	0.684	1.01	2.458	0.677

Average Gas Production Rate @ STP	
Two-phase System, L/d	Conventional System, L/d
2.665	0.639
Standard Deviation	
0.527	0.079

Run 1: Blended Primary Sludge

4/18

Date	Chemical Oxygen Demand, mg/L Total Sample				Chemical Oxygen Demand, mg/L Centrifuged Supernatant			
	feed	Reactor #1	Reactor #2	Reactor #3	feed	Reactor #1	Reactor #2	Reactor #3
23-Sep								
24-Sep								
25-Sep	39100	38288	17418	19497	4866	5950	6543	10546
26-Sep								
27-Sep								
28-Sep								
29-Sep								
30-Sep								
01-Oct								
02-Oct	41577	34449	24733	32300	6118	6649	6305	12321
03-Oct								
04-Oct								
05-Oct								
06-Oct								
07-Oct								
08-Oct								
09-Oct	30619	33645	20250	32901	4765	6258	6480	12068
10-Oct								
11-Oct								
12-Oct								
13-Oct								
14-Oct								
15-Oct								
16-Oct		35022	25615	34638		7383	6078	11514

Run 1: Blended Primary Sludge

5/18

Date	Chemical Oxygen Demand, mg/L Centrifuged filtrate				Alkalinity, mg/L as Calcium Carbonate			
	Feed	Reactor #1	Reactor #2	Reactor #3	Feed	Reactor #1	Reactor #2	Reactor #3
23-Sep								
24-Sep								
25-Sep	34234	32338	10875	8951	1875	2086	5249	9315
26-Sep								
27-Sep								
28-Sep								
29-Sep								
30-Sep								
01-Oct								
02-Oct	35460	27800	18428	19980	1431	1686	5539	10275
03-Oct								
04-Oct								
05-Oct								
06-Oct								
07-Oct								
08-Oct								
09-Oct	25854	27387	13770	20833	1770	1865	5387	11236
10-Oct								
11-Oct								
12-Oct								
13-Oct								
14-Oct								
15-Oct								
16-Oct		27639	19537	23124		2090	5168	10541

Run 1: Blended Primary Sludge

6/18

Date	Volatile Acids, mg/L as Acetic Acid			Digester Gas Analysis % CH ₄ , %CO ₂ , %N ₂ in off-gas	
	Reactor #1	Reactor #2	Reactor #3	Reactors #1 and #2	Reactor #3
23-Sep					
24-Sep					
25-Sep					
26-Sep					
27-Sep					
28-Sep					
29-Sep					
30-Sep					
01-Oct					
02-Oct				45.67,49.07,5.26	26.67,70.23,3.1
03-Oct					
04-Oct					
05-Oct					
06-Oct					
07-Oct					
08-Oct				50.33,47.02,2.65	33.72,64.11,2.17
09-Oct	28419	14406	12667		
10-Oct					
11-Oct					
12-Oct					
13-Oct					
14-Oct					
15-Oct				48.24,44.75,7.01	33.25,63.71,2.77
16-Oct	38364	16793	12570		

Run 1: Blended Primary Sludge

1/18

Date	Comments
23-Sep	Fed 5g Sodium Bicarb /266mL feed to Reactor 3
24-Sep	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 5g/266 feed to Reactor 3
25-Sep	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 5g/266 feed to Reactor 3 Masterflex pump broken so began gravity feeding
26-Sep	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 5g/266 feed to Reactor 3 ** ADAM MADE WEISS ARRIVED **
27-Sep	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 5g/266 feed to Reactor 3
28-Sep	Fed 1.65g Sodium Bicarb /266mL feed to Reactor 2; 5g/266 feed to Reactor 3
29-Sep	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 5g/266 feed to Reactor 3
30-Sep	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 5g/266 feed to Reactor 3
01-Oct	Fed 1g Sodium Bicarb /266mL feed to Reactor 2; 4g/266 to Reactor 3
02-Oct	Fed 4g Sodium Bicarb /266mL feed to Reactor 3
03-Oct	Fed 0.5g Sodium Bicarb /266mL feed to Reactor 2; 4.5g/266 feed to Reactor 3
04-Oct	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 5g/266 feed to Reactor 3
05-Oct	Fed 3g Sodium Bicarb /266mL feed to Reactor 2; 5g/266 feed to Reactor 3 Foaming in Reactors 2 and 3
06-Oct	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 5g/266 feed to Reactor 3 Excessive foaming in Reactor 2
07-Oct	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 4.5g/266 feed to Reactor 3
08-Oct	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 4g/266 feed to Reactor 3
09-Oct	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 4g/266 feed to Reactor 3 Mixer 1 was left on all night, therefore temperature in Reactor 1 was raised
10-Oct	Foaming in Reactor 1
11-Oct	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 4.5g/266 feed to Reactor 3
12-Oct	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 4.5g/266 feed to Reactor 3 Mixer 1 left on a higher mixing speed, therefore temperature rise in Reactor 1
13-Oct	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 4.5g/266 feed to Reactor 3
14-Oct	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 4.5g/266 feed to Reactor 3
15-Oct	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 4.5g/266 feed to Reactor 3
16-Oct	End of run; Began feeding Run 2 sludge on 10/17; Alk run 6 hrs after sampling

Appendix B-3.

Daily Operating Data during Homogenized Feed Study of Phase II

Run 2: Raw Primary Sludge; 20 day detention time; 16-day run; total phase 44 days

1/2R

Date	Time ----		Interval Time, days	Cumulative Time, days	Temperature, degrees Celsius -----				Barometric Pressure, mm Hg
	hour	minutes			Room	Reactor #1	Reactor #2	Reactor #3	
14-Nov	10	17			33.5	35	35	35.1	744.5
15-Nov	12	40	1.10	1.10	32.5	34	34.2	34	737.3
16-Nov	11	45	0.96	2.06	31.5	34	33.5	33.2	736.1
17-Nov	8	30	0.86	2.93	33	35.5	35	35	737.3
18-Nov	8	32	1.00	3.93	35	36.2	36	36	740.4
19-Nov	9	30	1.04	4.97	36.5	36.8	36.2	37	737.4
20-Nov	8	30	0.96	5.93	35.7	35.7	36	36	739.9
21-Nov	7	45	0.97	6.89	35.7	36.7	36.7	36.7	737.0
22-Nov	9	30	1.07	7.97	34.1	34.2	35.1	34.7	733.9
23-Nov	9	15	0.99	8.96	33.8	35.7	35.3	34.5	742.1
24-Nov	8	45	0.98	9.94	34	35	35	35	743.0
25-Nov	10	18	1.06	11.00	33.8	35	35	34.8	739.2
26-Nov	10	0	0.99	11.99	33.8	34.5	34.7	33.5	742.9
27-Nov	7	40	0.90	12.89	34	35.6	35.2	34.5	742.0
28-Nov	9	15	1.07	13.96	34.1	34.9	35.4	34.8	739.8
29-Nov	8	0	0.95	14.90	34.2	36	35.5	35.5	741.0

Run 2: Raw Primary Sludge

2/2R

Date	pH				Net-Test Meter Readings		Gas Production	
	Feed	Reactor #1	Reactor #2	Reactor #3	Two-phase system, L	Conventional System, cf	Two-phase system, L	Conventional System, cf
14-Nov	5.55	5.3	6.96	7.18	362.758	1.1888		
15-Nov	5.4	5.12	6.9	7.17	366.627	1.287	3.869	0.0982
16-Nov	5.15	5.13	7	7.18	369.975	1.3655	3.348	0.0785
17-Nov	6	5.25	6.96	7.13	372.775	1.425	2.8	0.0595
18-Nov	5.58	5.39	6.94	7.1	376.152	1.507	3.377	0.082
19-Nov	5.48	5.38	7	7.09	381.5	1.6145	5.348	0.1075
20-Nov	5.23	5.31	6.98	7.04	384.75	1.7472	3.25	0.1327
21-Nov	5.72	5.4	7.07	7.11	388.885	1.858	4.135	0.1108
22-Nov	5.81	5.53	7.05	7.1	392.835	1.9723	3.95	0.1143
23-Nov	5.6	5.45	7.11	7.13	396.768	2.076	3.933	0.1037
24-Nov	5.52	5.37	7.04	7.03	401.84	2.189	5.072	0.113
25-Nov	5.3	5.39	7.02	7.05	406.942	2.3165	5.102	0.1275
26-Nov	5.14	5.31	7.21	7.04	411.046	2.4283	4.104	0.1118
27-Nov	5.42	5.25	7.15	7.04	416.438	2.522	5.392	0.0937
28-Nov	5.41	5.3	7.22	7.02	422.035	2.6295	5.597	0.1075
29-Nov	5.4	5.3	7.13	7.02	426.31	2.7268	4.275	0.0973

Run 2: Raw Primary Sludge

3/2R

Date	Gas Production @ STP		Interval Time, days	Gas Production Rate @ STP		
	Two-phase System, L	Conventional System, L		Two-phase System, L/d	Conventional System, L/d	
14-Nov						
15-Nov	3.354	2.411	1.10	3.051	2.193	
16-Nov	2.907	1.931	0.96	3.023	2.007	
17-Nov	2.424	1.459	0.86	2.803	1.687	
18-Nov	2.916	2.005	1.00	2.912	2.003	
19-Nov	4.577	2.606	1.04	4.400	2.505	
20-Nov	2.798	3.236	0.96	2.920	3.376	
21-Nov	3.546	2.691	0.97	3.661	2.778	
22-Nov	3.391	2.779	1.07	3.161	2.590	
23-Nov	3.418	2.552	0.99	3.453	2.579	
24-Nov	4.410	2.782	0.98	4.504	2.841	
25-Nov	4.416	3.125	1.06	4.148	2.936	
26-Nov	3.570	2.754	0.99	3.615	2.789	
27-Nov	4.682	2.304	0.90	5.186	2.552	
28-Nov	4.844	2.635	1.07	4.544	2.472	
29-Nov	3.704	2.388	0.95	3.908	2.519	
						Average Gas Production Rate @ STP

						Two-phase Conventional
						System, L/d System, L/d

						4.280 2.653
						Standard Deviation

						0.546 0.179

Run 2: Raw Primary Sludge

4/2R

Date	Chemical Oxygen Demand, mg/L Total Sample				Chemical Oxygen Demand, mg/L Centrifuged Supernatant			
	feed	Reactor #1	Reactor #2	Reactor #3	feed	Reactor #1	Reactor #2	Reactor #3
14-Nov	36397	25549	28596	46459	7290	5916	7334	22-Apr
15-Nov								
16-Nov								
17-Nov								
18-Nov								
19-Nov								
20-Nov	35336	23994	22553	27358	5371	6448	4772	7125
21-Nov								
22-Nov								
23-Nov								
24-Nov								
25-Nov								
26-Nov								
27-Nov								
28-Nov								
29-Nov	41635	33004	18526	22935	6671	7591	1726	6047

Run 2: Raw Primary Sludge

5/2R

Date	Chemical Oxygen Demand, mg/L Centrifuged filtrate				Alkalinity, mg/L as Calcium Carbonate			
	Feed	Reactor #1	Reactor #2	Reactor #3	Feed	Reactor #1	Reactor #2	Reactor #3
14-Nov	29107	19633	21262	40868	1850	1761	4465	7188
15-Nov								
16-Nov								
17-Nov								
18-Nov								
19-Nov								
20-Nov	29965	17546	17781	20233	1760	2084	4949	6991
21-Nov								
22-Nov								
23-Nov								
24-Nov								
25-Nov								
26-Nov								
27-Nov								
28-Nov								
29-Nov	34964	25413	16800	16888				

Run 2: Raw Primary Sludge

6/2R

Date	Volatile Acids, mg/L as Acetic Acid			Digester Gas Analysis % CH ₄ , %CO ₂ , %N ₂ in off-gas	
	Reactor #1	Reactor #2	Reactor #3	Reactors #1 and #2	Reactor #3
14-Nov					
15-Nov					
16-Nov					
17-Nov					
18-Nov					
19-Nov					
20-Nov	14366	15916	17153		
21-Nov					
22-Nov					
23-Nov					
24-Nov					
25-Nov					
26-Nov					
27-Nov					
28-Nov					
29-Nov	1222	860	4185	59.61,38.56,1.33	66.22,32.61,1.17

Run 2: Raw Primary Sludge

1/28

Date	Comments
14-Nov	Alkalinities and COD values from 11/13
15-Nov	Fed 1.5g Sodium Bicarb / 100mL feed to Reactor 2
16-Nov	
17-Nov	
18-Nov	
19-Nov	
20-Nov	
21-Nov	
22-Nov	
23-Nov	
24-Nov	
25-Nov	
26-Nov	
27-Nov	
28-Nov	
29-Nov	Began feeding Reactors 2 and 3 at 10-day HRT with some raw feed

Appendix B-4.

Daily Operating Data during Primary Sludge Feed Study of Phase II

Run 2: Blended Primary Sludge; 20-day detention time; 28-day run

1/28

Date	Time		Interval Time, days	Cumulative Time, days	Temperature, degrees Celsius				Barometric Pressure, mm Hg
	hour	minutes			Room	Reactor #1	Reactor #2	Reactor #3	
21-Oct	8	45			35.5	35.7	36	35.7	744.1
22-Oct	7	25	0.94	0.94	34	35.2	34.5	35.2	740
23-Oct	8	10	1.03	1.98	34.2	35.8	35.5	35.2	741.8
24-Oct	8	23	1.01	2.98	34.5	35.2	35	35	741.6
25-Oct	10	10	1.07	4.06	34	35	35.2	35	740.4
26-Oct	8	43	0.98	5.04	34	35	34.5	34.5	738.8
27-Oct	8	54	1.01	6.05	34.2	34.5	35	35	737.6
28-Oct	8	28	0.98	7.03	34.8	36	35.2	35	736.0
29-Oct	8	33	1.00	8.03	34.5	35.8	36	35.6	745.9
30-Oct	9	22	1.03	9.07	34.5	35.5	35	35.2	746.7
31-Oct	7	49	0.94	10.00	34	35.2	35	34.3	740.6
01-Nov	9	0	1.05	11.05	34	35	34.5	35	749.9
02-Nov	8	50	0.99	12.05	34	35	34	34	753.8
03-Nov	8	30	0.99	13.03	34.8	35.2	35	35	749.8
04-Nov	8	32	1.00	14.03	34.5	35.2	35.5	35.5	745.2
05-Nov	12	13	1.15	15.19	35	36	35.8	35.8	739.3
06-Nov	9	0	0.87	16.05	34.5	36.5	36	36	738.1
07-Nov	9	56	1.04	17.09	34.7	35	35.5	35.5	737.4
08-Nov	9	28	0.98	18.07	34	36	35.8	35.3	736.5
09-Nov	10	45	1.06	19.13	33.2	37.2	35.7	35.3	749.3
10-Nov	10	5	0.97	20.10	33	35	35	35	750.2
11-Nov	8	30	0.93	21.03	34.5	35	35.5	35	749.5
12-Nov	8	20	0.99	22.02	34.5	35.7	35.7	35.3	753.1
13-Nov	9	9	1.03	23.06	34	35.2	35	34.5	754.0

Run 2: Blended Primary Sludge

2/28

Date	pH				Net-test Meter Readings		Gas Production	
	Feed	Reactor #1	Reactor #2	Reactor #3	Two-phase system, L	Conventional System, cf	Two-phase system, L	Conventional System, cf
21-Oct	5.6	5.41	7.01	7.02	279.833	0.6377		
22-Oct	5.77	5.5	7.07	7.04	282.785	0.7079	2.952	0.0702
23-Oct	5.75	5.47	7.03	7.07	286.045	0.7685	3.26	0.0606
24-Oct	5.7	5.4	7	7.02	289.41	0.8072	3.365	0.0387
25-Oct	5.62	5.45	7.1	7.1	292.16	0.8463	2.75	0.0391
26-Oct	5.62	5.42	7.07	7.08	295.58	0.8858	3.42	0.0395
27-Oct	5.7	5.26	7.11	7.12	299.16	0.9228	3.58	0.037
28-Oct	6.1	5.52	7	7.1	302.627	0.957		
29-Oct	5.85	5.46	7.03	7.08	305.713	0.9947	3.086	0.0377
30-Oct	5.5	5.4	7.05	7.04	309.4	1.052	3.687	0.0573
31-Oct	5.1	5.32	7.07	7.09	313.25	1.0983	3.85	0.0463
01-Nov	6.4	5.25	7.17	7.11	316.99	1.1413	3.74	0.043
02-Nov	6.37	5.23	7.25	7.08	320.205	1.201	3.215	0.0597
03-Nov	6.02	5.25	7.2	7.25	323.61	1.2652	3.405	0.0642
04-Nov	5.78	5.4	7.06	7.12	327.26	1.3198	3.65	0.0546
05-Nov	5.02	5.22	6.99	7.09	332.17	1.4095	4.91	0.0897
06-Nov	5.43	5.26	7.32	7.35	335.487	1.4732	3.317	0.0637
07-Nov	5.43	5.03	7.03	7.12	339.09	1.565	3.603	0.0918
08-Nov	5.48	5.18	7.02	7.13	342.485	1.6626	3.395	0.0976
09-Nov	5.52	5.28	7.01	7.25	346.73	1.7567	4.245	0.0941
10-Nov	5.52	5.38	7	7.28	349.645	1.8444	2.915	0.0877
11-Nov	5.3	5.38	6.98	7.3	352.575	1.9262	2.93	0.0818
12-Nov	6	5.38	6.97	7.31	355.97	2.016	3.395	0.0898
13-Nov	5.6	5.27	7.09	7.33	359.273	2.0953	3.303	0.0793

Run 2: Blended Primary Sludge

3/28

Date	Gas Production @ STP		Interval Time, days	Gas Production Rate @ STP		
	Two-phase System, L	Conventional System, L		Two-phase System, L/d	Conventional System, L/d	
21-Oct						
22-Oct	2.556	1.721	0.94	2.707	1.823	
23-Oct	2.828	1.489	1.03	2.742	1.444	
24-Oct	2.915	0.950	1.01	2.889	0.941	
25-Oct	2.383	0.959	1.07	2.218	0.893	
26-Oct	2.953	0.966	0.94	3.143	1.028	
27-Oct	3.088	0.904	1.01	3.064	0.897	
28-Oct			0.98			
29-Oct	2.689	0.930	1.00	2.680	0.927	
30-Oct	3.216	1.416	1.03	3.110	1.369	
31-Oct	3.336	1.136	0.94	3.567	1.215	
01-Nov	3.282	1.069	1.05	3.128	1.018	
02-Nov	2.836	1.491	0.99	2.856	1.502	
03-Nov	2.977	1.589	0.99	3.018	1.612	
04-Nov	3.178	1.346	1.00	3.173	1.344	
05-Nov	4.234	2.190	1.15	3.671	1.899	
06-Nov	2.860	1.556	0.87	3.303	1.796	
07-Nov	3.102	2.238	1.04	2.986	2.154	
08-Nov	2.926	2.382	0.98	3.001	2.443	
09-Nov	3.732	2.343	1.06	3.524	2.212	
10-Nov	2.567	2.187	0.97	2.641	2.250	
11-Nov	2.566	2.028	0.93	2.747	2.172	
12-Nov	2.987	2.237	0.99	3.008	2.253	
13-Nov	2.914	1.981	1.03	2.818	1.916	
						Average Gas Production Rate @ STP

						Two-phase System, L/d
						Conventional System, L/d

						2.803
						2.148

						Standard Deviation

						0.134
						0.138

Run 2: Blended Primary Sludge

4/28

Date	Chemical Oxygen Demand, mg/L Total Sample				Chemical Oxygen Demand, mg/L Centrifuged Supernatant			
	Feed	Reactor #1	Reactor #2	Reactor #3	Feed	Reactor #1	Reactor #2	Reactor #3
21-Oct								
22-Oct								
23-Oct	40040	36826	30694	33957	4315	6262	6863	13794
24-Oct	38804				5375			
25-Oct								
26-Oct								
27-Oct								
28-Oct								
29-Oct								
30-Oct	53645	53157	25948	39010	9830	9743	6569	13819
31-Oct								
01-Nov								
02-Nov								
03-Nov								
04-Nov								
05-Nov								
06-Nov	40886	44708	26802	33353	4891	8559	6096	12140
07-Nov								
08-Nov								
09-Nov								
10-Nov								
11-Nov								
12-Nov								
13-Nov	46459	36397	25549	28596	5591	7290	5916	7334

Run 2: Blended Primary Sludge

5/28

Date	Chemical Oxygen Demand, mg/L Centrifuged filtrate				Alkalinity, mg/L as Calcium Carbonate			
	Feed	Reactor #1	Reactor #2	Reactor #3	Feed	Reactor #1	Reactor #2	Reactor #3
21-Oct								
22-Oct								
23-Oct	35725	30564	23831	20163	1579	1717	5533	10008
24-Oct	33429							
25-Oct								
26-Oct								
27-Oct								
28-Oct								
29-Oct								
30-Oct	43815	43414	19379	25191				
31-Oct					1514	2600	5619	9273
01-Nov								
02-Nov								
03-Nov								
04-Nov								
05-Nov								
06-Nov	35995	36149	20706	21213	1727	2137	5551	9383
07-Nov								
08-Nov								
09-Nov								
10-Nov								
11-Nov								
12-Nov								
13-Nov	40868	29107	19633	21262	1850	1761	4465	7188

Run 2: Blended Primary Sludge

6/28

Date	Volatile Acids, mg/L as Acetic Acid			Digester Gas Analysis % CH ₄ , %CO ₂ , %N ₂ in off-gas	
	Reactor #1	Reactor #2	Reactor #3	Reactors #1 and #2	Reactor #3
21-Oct					
22-Oct					
23-Oct	32140	18285	16657		
24-Oct					
25-Oct					
26-Oct					
27-Oct					
28-Oct					
29-Oct					
30-Oct	14346	19075	18682		
31-Oct					
01-Nov					
02-Nov					
03-Nov					
04-Nov					
05-Nov					
06-Nov		3810	1978		
07-Nov					
08-Nov					
09-Nov					
10-Nov					
11-Nov					
12-Nov					
13-Nov	10760	14609	14598		

Run 2: Blended Primary Sludge

7/28

Date	Comments
21-Oct	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 4.5g/266 feed to Reactor 3
22-Oct	Fed 1.3g Sodium Bicarb /266mL feed to Reactor 2; 4g/266 feed to Reactor 3
23-Oct	
24-Oct	Fed 1.5g Sodium Bicarb /266mL feed to Reactor 2; 4g/266 feed to Reactor 3 feed COD values from particle size analysis run 10/23
25-Oct	Fed 1.3g Sodium Bicarb /266mL feed to Reactor 2; 4g/266 feed to Reactor 3
26-Oct	Fed 1.3g Sodium Bicarb /266mL feed to Reactor 2; 4g/266 feed to Reactor 3
27-Oct	Daylight Savings Time; therefore, 1 hour was added to the interval time formula Replaced septa in Reactors 1 and 2 gas line
28-Oct	Fed 1.3g Sodium Bicarb /266mL feed to Reactor 2
29-Oct	Fed 1.3g Sodium Bicarb /266mL feed to Reactor 2
30-Oct	Fed 1.3g Sodium Bicarb /266mL feed to Reactor 2; 4g/266 feed to Reactor 3 Reactor 3 cent. super. COD disturbed; therefore, higher than normal
31-Oct	
01-Nov	Fed 1.3g Sodium Bicarb /266mL feed to Reactor 2; 4g/266 feed to Reactor 3
02-Nov	Fed 1.3g Sodium Bicarb /266mL feed to Reactor 2; 4g/266 feed to Reactor 3
03-Nov	
04-Nov	
05-Nov	Fed 1.3g Sodium Bicarb /266mL feed to Reactor 2
06-Nov	
07-Nov	Mixer 1 was accidently left off all night
08-Nov	No 10 p.m. feeding for Reactor 1 due to feed still cold
09-Nov	Mixer 1 was left on all night, therefore temperature in Reactor 1 was raised
10-Nov	
11-Nov	
12-Nov	
13-Nov	End of run; Meter 3 has been measuring more gas than actual due to the fact fact that the water level in the meter was low

Appendix B-5.

Daily Operating Data during Primary Sludge Feed Study of Phase III

Run 3: Raw Primary Sludge; 10 day detention time; 21-day run 1/3R

Date	Time		Interval Time, days	Cumulative Time, days	Temperature, degrees Celsius				Barometric Pressure, mm Hg
	hour	minutes			Room	Reactor #1	Reactor #2	Reactor #3	
03-Dec	8	10			34	36	35	35	741.5
04-Dec	8	25	1.01	1.01	34	35	34.8	34.8	747.3
05-Dec	9	30	1.05	2.06	33.5	35	35.2	34.8	748.7
06-Dec	10	20	1.03	3.09	35	35.5	35.5	35	747.6
07-Dec	10	5	0.99	4.08	34.8	35.5	35	34.5	742.0
08-Dec	10	0	1.00	5.08	33.8				739.6
09-Dec	8	10	0.92	6.00	33	35.5	35.7	35	741.8
10-Dec	10	15	1.09	7.09	32.8	34.9	35.2	35	746.7
11-Dec	10	30	1.01	8.10	33.7	35.1	35.9	34.9	735.8
12-Dec	8	45	0.93	9.02	33.9	34.8	35.2	35.3	745.8
13-Dec	10	0	1.05	10.08	34.8	34.8	35.2	35.1	751.5
14-Dec	9	40	0.99	11.06	33.3	34.1	35.7	34.6	742.2
15-Dec	8	49	0.96	12.03	34	34.7	35.6	35.3	743.2
16-Dec	9	5	1.01	13.04	33.9	35.6	35.5	35.2	744.9
17-Dec	9	0	1.00	14.03	34	36.1	35.6	35.2	741.0
18-Dec	9	5	1.00	15.04	33.9	36.3	35.7	35.2	743.1
19-Dec	9	42	1.03	16.06	33.8	35.7	35.5	35.1	740.0

Run 3: Raw Primary Sludge

2/3R

Date	pH				Wet-Test Meter Readings		Gas Production	
	Feed	Reactor #1	Reactor #2	Reactor #3	Two-phase system, L	Conventional System, cf	Two-phase system, L	Conventional System, cf
03-Dec	5.48	5.3	7.1	7.03	453.89	3.3908		
04-Dec	5.27	5.34	7.12	6.94	460.78	3.5557	6.89	0.1649
05-Dec	5.46	5.17	7.14	6.91	466.41	3.6999	5.63	0.1442
06-Dec	5.48	5.24	6.99	6.82	474.6	3.8448		
07-Dec	4.98	5.32	7.01	6.92	481.815	3.9887	7.215	0.1439
08-Dec					488.02	4.132	6.205	0.1433
09-Dec	5.52	5.22	7	7.02	494.045	4.2593	6.025	0.1273
10-Dec	5.57	5.41	7.13	7.03	501.818	4.4059	7.773	0.1466
11-Dec		5.37	7.02	6.98	508.716	4.5623	6.898	0.1564
12-Dec	5.71	5.38	7.01	6.96	514.77	4.7205	6.054	
13-Dec	5.52	5.37	7.02	6.94	521.003	4.8863	6.233	0.1658
14-Dec	5.56	5.4	6.94	6.95	528.325	5.0557	7.322	0.1694
15-Dec	5.66	5.42	6.94	6.96	535.785	5.2245	7.46	0.1688
16-Dec	5.61	5.46	7.01	6.93	542.236	5.393	6.451	0.1685
17-Dec	5.63	5.32	6.99	6.89	548.132	5.5856	5.896	0.1926
18-Dec	5.46	5.24	7.07	6.89	554.216	5.7959	6.084	0.2103
19-Dec	5.62	5.24	6.97	6.86	560.745	6.0042	6.529	0.2083

Run 3: Raw Primary Sludge

3/3R

Date	Gas Production @ STP		Interval Time, days	Gas Production Rate @ STP		
	Two-phase System, L	Conventional System, L		Two-phase System, L/d	Conventional System, L/d	
03-Dec						
04-Dec			1.01			
05-Dec			1.05			
06-Dec			1.03			
07-Dec	6.248	3.529	0.99	6.314	3.566	
08-Dec	5.374	3.514	1.00	5.392	3.527	
09-Dec	5.247	3.140	0.92	5.681	3.399	
10-Dec	6.818	3.642	1.09	6.274	3.351	
11-Dec	5.945	3.817	1.01	5.884	3.778	
12-Dec	5.285		0.93	5.781		
13-Dec	5.467	4.118	1.05	5.196	3.914	
14-Dec	6.374	4.176	0.99	6.463	4.235	
15-Dec	6.488	4.157	0.96	6.726	4.310	
16-Dec	5.625	4.161	1.01	5.563	4.115	
17-Dec	5.112	4.729	1.00	5.130	4.746	
18-Dec	5.292	5.188	1.00	5.274	5.162	
19-Dec	5.657	5.111	1.03	5.516	4.983	
						Average Gas Production Rate @ STP

						Two-phase Conventional
						System, L/d System, L/d

						5.386 4.964
						Standard Deviation

						0.159 0.171

Run 3: Raw Primary Sludge

4/3R

Date	Chemical Oxygen Demand, mg/L Total Sample				Chemical Oxygen Demand, mg/L Centrifuged Supernatant			
	Feed	Reactor #1	Reactor #2	Reactor #3	Feed	Reactor #1	Reactor #2	Reactor #3
03-Dec								
04-Dec								
05-Dec								
06-Dec								
07-Dec								
08-Dec								
09-Dec								
10-Dec								
11-Dec								
12-Dec								
13-Dec								
14-Dec								
15-Dec								
16-Dec								
17-Dec								
18-Dec								
19-Dec		42977	19499	25309	8899	3649	1043	

Run 3: Raw Primary Sludge

5/3R

Date	Chemical Oxygen Demand, mg/L Centrifuged Filtrate				Alkalinity, mg/L as Calcium Carbonate			
	Feed	Reactor #1	Reactor #2	Reactor #3	Feed	Reactor #1	Reactor #2	Reactor #3
03-Dec								
04-Dec					1613	2145	4758	5299
05-Dec								
06-Dec								
07-Dec								
08-Dec								
09-Dec								
10-Dec								
11-Dec								
12-Dec								
13-Dec								
14-Dec								
15-Dec								
16-Dec								
17-Dec								
18-Dec								
19-Dec		34878	15850	24266		2161	3564	3986

Run 3: Raw Primary Sludge

6/3R

Date	Volatile Acids, mg/L as Acetic Acid			Digester Gas Analysis % CH ₄ , %CO ₂ , %N ₂ in off-gas	
	Reactor #1	Reactor #2	Reactor #3	Reactors #1 and #2	Reactor #3
03-Dec				27.39,41.25,31.36	60.16,30.21,1.63
04-Dec					
05-Dec					
06-Dec					
07-Dec					
08-Dec					
09-Dec					
10-Dec					
11-Dec					
12-Dec					
13-Dec					
14-Dec					
15-Dec					
16-Dec					
17-Dec					
18-Dec					
19-Dec	1841	3557	4510	12.62,26.77,.61	75.78,23.71,.51

Run 3: Raw Primary Sludge

7/3R

Date	Comments
03-Dec	Fed 4g Sodium Bicarb /266mL feed to Reactor 2; 6g/266 to Reactor 3 Leaks in two-phase system repaired based on gas analysis
04-Dec	New COD Standard, Alkalinities run
05-Dec	Reset gas meter readings
06-Dec	
07-Dec	
08-Dec	
09-Dec	
10-Dec	
11-Dec	
12-Dec	Reset reactor 3 gas meter
13-Dec	
14-Dec	
15-Dec	
16-Dec	
17-Dec	
18-Dec	
19-Dec	

Appendix B-6.

Daily Operating Data during Homogenized Feed Study of Phase III

Run 3: Blended Primary Sludge; 10 day detention time; 11-day run; total phase 32 days

1/38

Date	Time		Interval Time, days	Cumulative Time, days	Temperature, degrees Celsius				Barometric Pressure, mm Hg
	hour	minutes			Room	Reactor #1	Reactor #2	Reactor #3	
21-Dec	9	40			34.8	35.3	35.2	35	750.5
22-Dec	9	5	0.98	0.98	33.9	34.6	34.5	34.1	746.8
23-Dec	9	22	1.01	1.99	33.9	35.9	35.5	34.8	741.6
24-Dec	9	10	0.99	2.98	33.9	35.8	35.5	35	737.6
25-Dec	7	28	0.93	3.91	32.5	35.7	35.5		743.7
26-Dec	7	45	1.01	4.92	34.8	35	34	34.8	745.2
27-Dec	8	50	1.05	5.97	34	36	35.5	35.5	744.2
28-Dec	8	32	0.99	6.95	33.7	35.5	35	35	742.8
29-Dec	7	35	0.96	7.91	34	35.5	35	35	737.0
30-Dec	8	5	1.02	8.93	33.7	36.2	35.8	35.4	743.2

Run 3: Blended Primary Sludge

2/38

Date	pH				Vet-Test Meter Readings		Gas Production	
	Feed	Reactor #1	Reactor #2	Reactor #3	Two-phase system, L	Conventional System, cf	Two-phase system, L	Conventional System, cf
21-Dec	5.56	5.23	6.97	6.92	573.227	6.3728		
22-Dec	5.63	5.37	6.98	6.9	579.325	6.5388	6.098	0.166
23-Dec	5.64	5.36	7.01	6.92	585.552	6.7322	6.227	0.1934
24-Dec	5.75	5.4	7.02	6.93	591.35	6.9048	5.798	0.1726
25-Dec	5.82	5.37	6.92		597	7.0765	5.65	0.1717
26-Dec	5.65	5.4	6.89	6.8	603.22	7.254	6.22	0.1775
27-Dec	5.48	5.25	6.97	6.83	609.34	7.4635	6.12	0.2095
28-Dec	5.18	5.28	6.9	6.83	614.86	7.656	5.52	0.1925
29-Dec	5.3	5.15	6.9	6.82	621.27	7.8467	6.41	0.1907
30-Dec	5.68	5.13	7.15	7.05	628.245	8.0532	6.975	0.2065

Run 3: Blended Primary Sludge

1/38

Date	Gas Production @ STP		Interval Time, days	Gas Production Rate @ STP		Average Gas Production Rate @ STP	
	Two-phase System, L	Conventional System, L		Two-phase System, L/d	Conventional System, L/d		
21-Dec							
22-Dec	5.331	4.110	0.98	5.463	4.212		
23-Dec	5.405	4.754	1.01	5.342	4.699		
24-Dec	5.006	4.220	0.99	5.048	4.256		
25-Dec	4.941	4.252	0.93	5.318	4.577		
26-Dec	5.410	4.372	1.01	5.347	4.321		
27-Dec	5.329	5.167	1.05	5.099	4.944		
28-Dec	4.803	4.743	0.99	4.863	4.803	5.852	4.918
29-Dec	5.528	4.658	0.96	5.756	4.849		
30-Dec	6.072	5.091	1.02	5.948	4.987	0.096	0.069

Run 3: Blended Primary Sludge

4/38

Date	Chemical Oxygen Demand, mg/L Total Sample				Chemical Oxygen Demand, mg/L Centrifuged Supernatant			
	Feed	Reactor #1	Reactor #2	Reactor #3	Feed	Reactor #1	Reactor #2	Reactor #3
21-Dec		42997	19499	25309		8099	3649	1043
22-Dec								
23-Dec								
24-Dec								
25-Dec								
26-Dec								
27-Dec								
28-Dec								
29-Dec								
30-Dec	42251	37252	21414	23610	6778	10366	1291	3160

Run 3: Blended Primary Sludge

5/3B

Date	Chemical Oxygen Demand, mg/L Centrifuged Filtrate				Alkalinity, mg/L as Calcium Carbonate			
	Feed	Reactor #1	Reactor #2	Reactor #3	Feed	Reactor #1	Reactor #2	Reactor #3
21-Dec		34898	15850	24266		2161	3564	3986
22-Dec								
23-Dec								
24-Dec								
25-Dec								
26-Dec								
27-Dec								
28-Dec								
29-Dec								
30-Dec	35473	26886	20123	20450	2335	2370	3595	3946

Run 3: Blended Primary Sludge

6/3B

Date	Volatile Acids, mg/L as Acetic Acid			Digester Gas Analysis % CH ₄ , %CO ₂ , %H ₂ in off-gas	
	Reactor #1	Reactor #2	Reactor #3	Reactors #1 and #2	Reactor #3
21-Dec				72.62,26.77,.61	75.78,23.71,.51
22-Dec					
23-Dec					
24-Dec					
25-Dec					
26-Dec					
27-Dec					
28-Dec					
29-Dec					
30-Dec	2359	1095	1905	61.61,35.75,2.64	62.26,35.58,2.16

Run 3: Blended Primary Sludge

7/38

Date	Comments
21-Dec	COD, Alkalinity, and Gas Analysis from 12/19
22-Dec	
23-Dec	
24-Dec	
25-Dec	
26-Dec	
27-Dec	
28-Dec	
29-Dec	
30-Dec	

APPENDIX C.

DATA USED IN CALCULATION OF GAS PRODUCTION RATE PER GRAM COD

The calculated values for the gas production rates are given in Table C-1. Data used in calculation of the gas production rates are also given in Table C-1. Incorrect gas meter readings during the 20-day phase (phase II) resulted in the gas production rate not being calculated. As stated in Chapter IV, the theoretical value for gas production per gram of COD destroyed is 0.35. The theoretical gas production rate reported in Table C-1 was calculated by multiplying the actual value for grams of COD destroyed by 0.35 L CH₄/g COD destroyed. The gas production rate per gram of COD applied was then calculated by dividing the theoretical gas production rate by the actual value for COD applied. The variability of the rates given in the table is due primarily to the highly variable COD of the influent feed used in the study.

The variability of the influent feed COD can be seen in column A of Table C-2. In order to calculate a reasonable average influent feed COD for each phase, the relatively small data set was expanded. Therefore, the relationships which existed between the influent feed COD and both the COD of the contents of the acidogenic reactor (reactor #1) and the COD of the centrate after the feed sludge had been centrifuged at 39100 G for 30 minutes, were utilized.

As can be seen in Table C-2, an average value was calculated for the relationship between the influent feed COD and reactor #1 COD for

Table C-1. Data used in calculation of the gas production rates in each system

Gas Production Rate per gram COD destroyed

Hydraulic Retention Time, d	Gas Production Rate, L/d					Influent COD, mg/L		Std Dev of Influent COD		Effluent COD, mg/L	
	Raw		Blended		Daily Feed, L	Raw	Blended	Raw	Blended	Raw	Blended
	COD	CH ₄	COD	CH ₄							
10-day											
Conv.	4.964	76	4.918	62	0.4	49467	41998		1613	25309	23610
Two Phase	5.306	73	5.852	62	0.444	49467	41098		1613	19499	21414
15-day											
Conv.	1.085	30	0.639	34	0.267	37701	37962		5782	27188	22300
Two Phase	2.54	40	2.665	50	0.267	37701	37962		5782	23005	24733
20-day											
Conv.	2.653	66	2.148	70	0.2	37855	39402		8157	22935	33353
Two Phase	4.28	60	2.803	70	0.211	37855	39402		8157	18526	26802

Hydraulic Retention Time, d	Gas Production, L CH ₄ /d		COD Destroyed, g/d		COD Applied, g/d		Percent COD Removed	
	Raw	Blended	Raw	Blended	Raw	Blended	Raw	Blended
10-day								
Conv.	3.773	3.049	9.663	6.995	19.79	16.44	49	43
Two Phase	3.873	3.628	13.306	8.740	21.96	18.25	61	48
15-day								
Conv.	0.326	0.217	3.341	1.512	10.60	10.14	32	15
Two Phase	1.016	1.333	4.458	3.532	10.60	10.14	42	35
20-day								
Conv.	1.751	1.504	2.984	1.210	7.57	7.88	39	15
Two Phase	2.568	1.962	4.078	2.659	7.99	8.31	51	32

Hydraulic Retention Time, d	Theoretical Gas Production, L CH ₄ /d		Gas Production, L CH ₄ /g COD applied		Gas Production, L CH ₄ /g COD destroyed	
	Raw	Blended	Raw	Blended	Raw	Blended
10-day						
Conv.	3.382	2.448	0.171	0.149	0.390	0.436
Two Phase	4.657	3.059	0.212	0.168	0.291	0.415
15-day						
Conv.	1.169	0.529	0.110	0.052	0.097	0.144
Two Phase	1.560	1.236	0.147	0.122	0.228	0.377

$$\text{COD Destroyed, g/d} = ((\text{Influent COD}(\text{Daily Feed}) + \text{Effluent COD}(\text{Daily Feed}))$$

$$- \text{Effluent COD}(\text{10})) / 1000)$$

Table C-2. Summary of data used to calculate average values for influent feed COD during each phase of the study

Detention Time,d R=Raw B=Blended	A Feed COD, mg/L	B Reactor #1 COD, mg/L	C Supernatant Feed COD,mg/L	D Average A/B	E Average A/C
10R		42977		1.151	6.62
10B	42251	37252	6778	1.042	6.23
15R	34019	39831	4941	1.151	6.62
	42067		6626	1.151	6.62
15B	39100	38282	4866	1.042	6.62
	41577	34449	6118	1.042	6.62
	30619	33645	4765	1.042	6.62
		35022		1.042	6.62
	40831		7828	1.042	6.62
20R	36397	25549	7290	1.151	7.1
	35336	23994	5371	1.151	7.1
	41635	33004	6671	1.151	7.1
	42364		4000	1.151	7.1
20B	40040	36826	4315	1.042	7.58
	38804		5375	1.042	7.58
	53645	53157	9830	1.042	7.58
	40886	44708	4891	1.042	7.58

each of the two feed studies, raw and blended. These values were calculated by dividing the average influent feed COD by the average reactor #1 COD for each of the two feed studies.

The relationship between the supernatant feed COD and the influent feed COD was also averaged due to the variability of the percentage of supernatant COD in the influent COD. An average value was calculated over the entire set of data for phase I (15-day HRT) while separate averages were calculated for each of the feed studies of phase II (20-day HRT) and the blended feed study of phase III (10-day HRT). Due

Table C-3. Expanded data set for influent feed COD including average overall feed COD and standard deviation

Detention Time, d R=Raw B=Blended	Feed COD, mg/L			Average Feed COD, mg/L	Standard Deviation
	As Analyzed	As Calculated from Reactor #1 COD D+B	As Calculated from Supernatant Feed COD E+C		
10R		49467		49467	
10B	42251	38817	42227	41098	1613
15R	34019	45845	32709	39701	5782
	42067		43864		
15B	39100	39690	32213	37962	7337
	41577	35896	40501		
	36619	35058	31544		
	40831	36493	51821		
20R	36397	29407	51759	37855	8157
	35336	27617	38134		
	41635	37988	47364		
	42364		28400		
20B	40040	38373	32708	39402	3053
	38604		40743		
	40686	46586	37074		

to insufficient data, the value assumed for phase III during the raw feed study was the same as calculated from the phase I (15-day HRT) data. Values for approximate influent COD were then calculated by multiplying the reactor #1 COD and the supernatant feed COD by the appropriate relationships shown in columns D and E of Table C-2. The expanded data set for influent feed COD is given in Table C-3, along with the average overall feed COD and standard deviation which were used in the calculations represented by Table C-1.

APPENDIX D.

SUMMARY OF DATA COLLECTED DURING PARTICLE SIZE FRACTIONATION STUDIES

The data collected for the particle size fractionation of feed sludges and reactor contents are given in Tables D-1 and D-2. Fractionation of samples was accomplished utilizing sedimentation followed by centrifugation. Each fraction represented a cumulative COD and contained the remaining COD in the sample. Each cumulative fraction was also represented by a maximum calculated equivalent diameter particle in the fraction. The last fraction, group III, represents the more soluble portion of each sample. A complete description of the particle size fractionation technique is given in Chapter IV. Notation used in Tables D-1 and D-2 was also summarized in Tables 11 and 13 in Chapter IV.

Incremental COD distributions were calculated by subtraction of adjacent cumulative data. The incremental data are presented in Tables D-3 and D-4 along with percentage of the total sample COD that each incremental COD fraction contains. Notation used in Tables D-3 and D-4 is summarized in Table 14 of Chapter IV.

The data contained in Tables D-1 thru D-4 were the result of fractionation of single samples at the end of each feed study of phases II and III. Therefore, the data are assumed approximate and was used to show trends while operating the anaerobic systems using different feed sludges. Comparisons between the two systems were also made based on the data obtained.

Table D-1. Summary of cumulative COD fractions of average feed sample and reactor contents during the primary sludge feed study of each phase

Sample Analyzed	Cumulative COD in indicated fraction, mg/L			
	Total	I	II	III
Primary Sludge	42000	12338	5884	5336
<u>10-day HRT</u>				
Conventional System	25309	13237	2658	1043
Two Phase System				
Acidogenic	42977	20248	9897	8099
Methanogenic	19499	13847	6011	3649
<u>20-day HRT</u>				
Conventional System	22945	20684	7384	5900
Two Phase System				
Acidogenic	33004	20506	8824	7591
Methanogenic	18526	15398	2320	1726

Table D-2. Summary of cumulative COD fractions of average feed sample and reactor contents during homogenized feed study of each phase

Sample Analyzed	Cumulative COD in indicated fraction, mg/L			
	Total	I	II	III
Homogenized Feed	41569	17228	7611	5835
<u>10-day HRT</u>				
Conventional System	23610	20134	4879	3160
Two Phase System				
Acidogenic	37252	32095	13461	10366
Methanogenic	21414	21330	2577	1291
<u>20-day HRT</u>				
Conventional System	33353	18848	14656	12140
Two Phase System				
Acidogenic	44708	18412	12210	8559
Methanogenic	26802	18639	7633	6096

Table D-3. Summary of incremental COD fractions of average feed sample and reactor contents during the primary sludge feed study of each phase

Sample Analyzed	Incremental COD in indicated fraction, mg/L (% of total)				
	Total	Total-I	I-II	II-III	III
Primary Sludge	42000	29662(71)	6454(15)	548(1)	5336(13)
<u>10-day HRT</u>					
Conventional System	25309	12072(48)	10579(42)	1615(6)	1043(4)
Two Phase System					
Acidogenic	42977	22729(53)	10351(24)	1798(4)	8099(19)
Methanogenic	19499	5652(29)	7836(40)	2362(12)	3649(19)
<u>20-day HRT</u>					
Conventional System	22945	2261(10)	13300(58)	1484(6)	5900(26)
Two Phase System					
Acidogenic	33004	12498(38)	11682(35)	1233(4)	7591(23)
Methanogenic	18526	3128(17)	13078(71)	594(3)	1726(9)

Table D-4. Summary of incremental COD fractions of average feed sample and reactor contents during homogenized feed study of each phase

Sample Analyzed	Incremental COD in indicated fraction, mg/L (% of total)				
	Total	Total-I	I-II	II-III	III
Homogenized Feed	41569	24341(59)	9617(23)	1476(4)	5835(14)
<u>10-day HRT</u>					
Conventional System	23610	3476(15)	15255(65)	1719(7)	3160(13)
Two Phase System					
Acidogenic	37252	5157(14)	18634(50)	3095(8)	10366(28)
Methanogenic	21414	84(<1)	18753(88)	1286(6)	1291(6)
<u>20-day HRT</u>					
Conventional System	33353	14505(43)	4192(13)	2516(8)	12140(36)
Two Phase System					
Acidogenic	44708	26296(59)	6202(14)	3651(8)	8559(19)
Methanogenic	26802	8163(30)	11006(41)	1537(6)	6096(23)

APPENDIX E.

SUMMARY OF ANALYSIS PERFORMED FOR INDIVIDUAL VOLATILE
ORGANIC ACIDS IN EACH REACTOR

Individual volatile organic acids concentrations of reactor contents were analyzed by gas chromatography. Results of the analysis and a computed value for total volatile organic acids as acetic acid are given for each reactor. Included in each set of data is the date on which the samples were taken and an abbreviated description of the study being performed at the time of sampling. For example, a sample was taken from the acidogenic reactor (reactor #1) on 10/9/86 during the homogenized feed study of phase I (15-day HRT) and abbreviated simply as 15d-H. Similarly, a sample was taken on 11/20/86 during the primary sludge feed study of phase II (20-day HRT) and abbreviated as 20d-P. All samples were analyzed at the end of the study with concentrations reported in mg/L.

The equation used to calculate the total volatile acids concentration as Acetic Acid was as follows:

$$\text{Total} = A + [P/74.1 + (IB + NB)/88.1 + (IV + NV)/132] * 60.1$$

where:

- A = Concentration of Acetic Acid, mg/L
- P = Concentration of Propionic Acid, mg/L
- IB = Concentration of Iso Butyric Acid, mg/L
- NB = Concentration of N-Butyric Acid, mg/L
- IV = Concentration of Iso Valeric Acid, mg/L

NV - Concentration of N-Valeric Acid, mg/L

Concentrations of Volatile Acids in Acidogenic Reactor (Reactor #1) in mg/L

Date	Run	Acetic	Propionic	Iso Butyric	N-Butyric	Iso Valeric	N-Valeric	Total, as Acetic
10/9	15d-H	24677	1036	348	3457	283	396	28419
10/16	15d-H	31181	1547	599	7438	484	506	38364
10/23	20d-H	23484	1414	673	9459	664	663	32140
10/30	20d-H	11160	547	359	3076	308	574	14346
11/13	20d-H	7994	610	260	2578	260	479	10760
11/20	20d-P	11496	784	371	2520	246	335	14366
11/29	20d-P	798	272	ND	289	16	ND	1222
12/19	10d-P	1625	ND	ND	295	22	10	1841
12/30	10d-H	2068	83	ND	295	18	34	2359

ND : Not Detected

Concentrations of Volatile Acids in Methanogenic Reactor (Reactor #2) in mg/L

Date	Run	Acetic	Propionic	Iso-Butyric	N-Butyric	Iso-Valeric	N-Valeric	Total, as Acetic
10/2	15d-H	11730	413	22	3305	160	151	14406
10/9	15d-H	13889	538	27	3386	188	128	15192
10/23	20d-H	14233	1342	87	4136	37	159	18285
10/30	20d-H	15417	870	49	4176	36	124	19675
11/6	20d-H	3658	140	ND	57	ND	ND	3855
11/13	20d-H	10463	2169	ND	3480	ND	35	14687
11/20	20d-P	12904	464	41	3805	ND	32	15246
11/29	20d-P	748	110	ND	33	ND	ND	891
12/19	10d-P	2938	715	ND	57	ND	ND	3710
12/30	10d-H	904	236	ND	ND	ND	ND	1140

ND : Not Detected

Concentrations of Volatile Acids in Conventional Reactor (Reactor #3) in mg/L

Date	Run	Acetic	Propionic	Iso-Butyric	N-Butyric	Iso-Valeric	N-Valeric	Total, as Acetic
10/2	15d-H	9462	708	287	3280	163	128	13928
10/9	15d-H	9352	855	359	3180	122	125	13893
10/23	20d-H	12157	1232	383	4355	282	126	18535
10/30	20d-H	15078	1114	337	3280	140	202	19821
11/6	20d-H	1179	700	97	62	61	275	1979
11/13	20d-H	13748	708	69	211	75	111	14892
11/20	20d-P	14624	1328	ND	1963	197	55	17157
11/29	20d-P	1201	866	77	3086	199	77	4496
12/19	10d-P	1421	597	86	3580	78	157	4829
12/30	10d-H	11	1815	ND	ND	ND	ND	1826

APPENDIX F.

SUMMARY OF RESULTS OF PHASE II PARTICLE SIZE
FRACTIONATION STUDIES

The fractionation of feed sludges and reactor contents was performed as described in Chapter IV. Data were collected at the end of study. A cumulative distribution of COD over various particle size fractions was obtained. The incremental COD concentration of each fraction was calculated from the cumulative values. The notation used for the cumulative data is given in Table 13 of Chapter IV, and for the incremental COD in each fraction in Table 14. Data collected for the cumulative COD distributions and the calculated incremental fractions COD for each sample are tabulated in Appendix D. Samples were collected only once for each study and therefore, values are assumed to represent trends in the anaerobic systems and not exact transformations of the COD.

Also, the fractionation technique used in the particle size distribution studies involving sedimentation followed by centrifugation was approximate due to inherent problems existing with centrifugation. Initial centrifugation of a sample will remove a percentage of all particle size fractions. However, the percentage of each fraction removed by the interaction of particles during centrifugation was assumed to be minimal. Relatively dilute samples were analyzed throughout the study and most of the large settleable particles were removed by sedimentation.

Both anaerobic treatment systems had been operating for 28 days at

a 20-day hydraulic retention time (HRT) prior to beginning the phase II, 20-day HRT study. Throughout phase II, the methanogenic reactor of the two-phase system operated at an HRT of 19 days and the acidogenic reactor at a one-day HRT. Therefore, the methanogenic reactor operated at an HRT of one day less than the conventional reactor. The difference in system HRTs was five percent (5 %) and was considered negligible.

The results of the particle size fractionation study are shown in Figures F-1 and F-2.

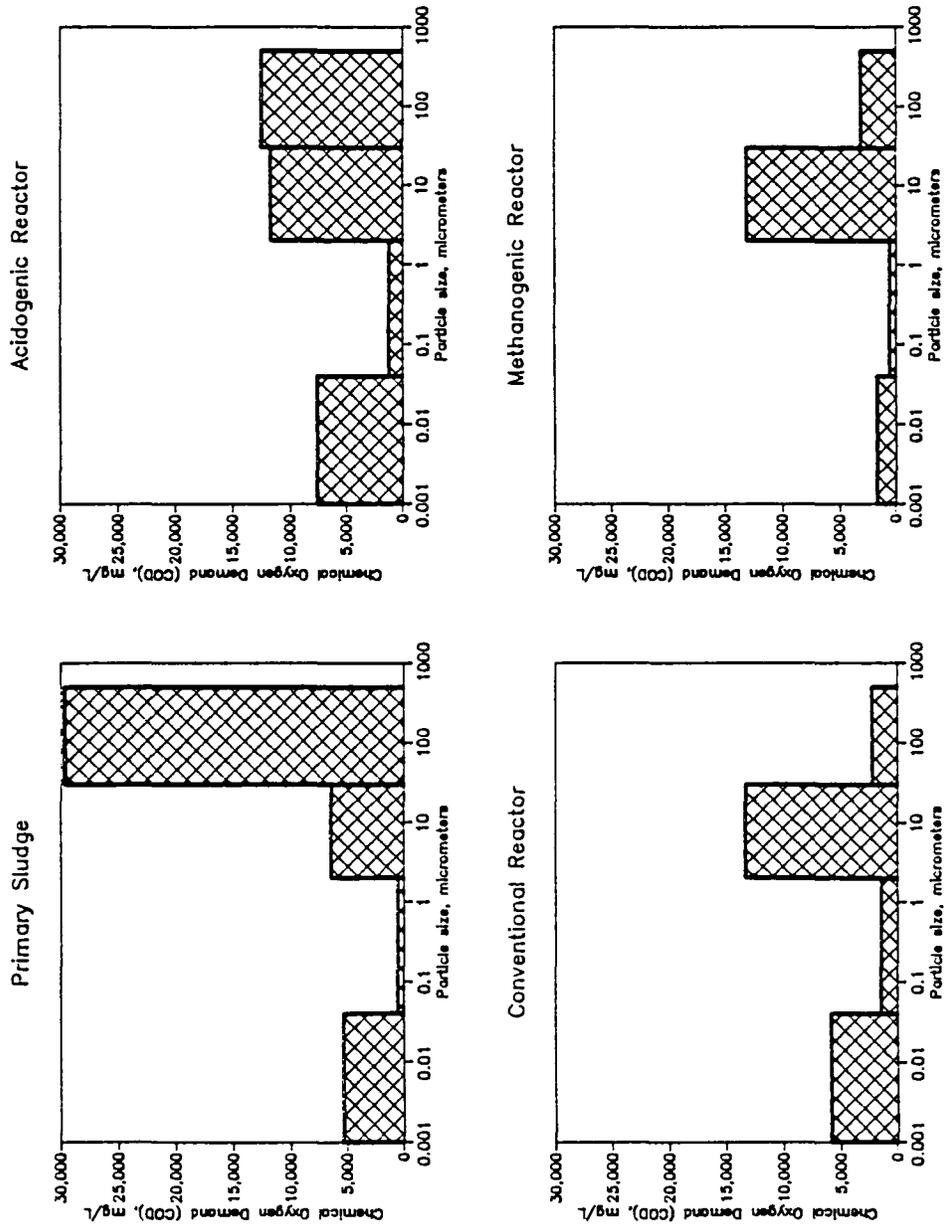


Figure F-1. Incremental COD distributions for samples taken during phase II, 20-day HRT, during the primary sludge feed study, with equivalent particle size diameters of each fraction defined in Table 20 of Chapter V. The fraction between 0.001 and 0.04 micrometers represents the more soluble portion of each sample

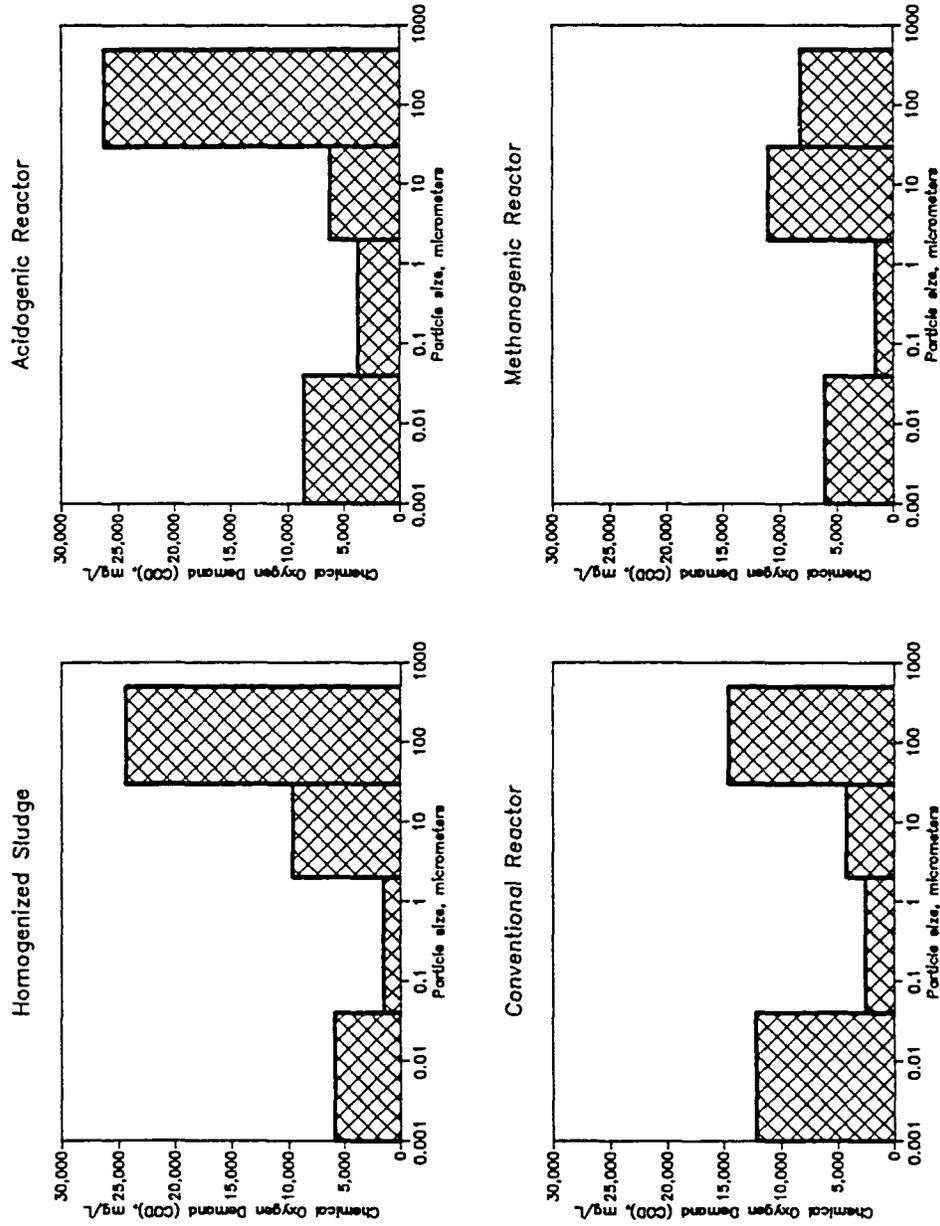


Figure F-2. Incremental COD distributions for samples taken during phase II, 20-day HRT, during the homogenized feed study, with equivalent particle size diameters of each fraction defined in Table 20 of Chapter V. The fraction between 0.001 and 0.04 micrometers represents the more soluble portion of each sample

APPENDIX G. DATA USED IN CALCULATION OF HYDROLYSIS RATE CONSTANTS

The calculated values for the hydrolysis rate constants for the acidogenic reactor of the two-phase system during both feed studies of phases II and III are given in Table G-1. Data used in calculation of the hydrolysis rate constants are also presented in Table G-1. An equation defining the rate of hydrolysis was given previously in Chapter III, equation 10, and is restated as follows:

$$F = \frac{F_o}{1 + k_h \Theta}$$

where:

k_h = first-order hydrolysis rate constant;

F_o = influent concentration of degradable particulate COD;

F = effluent concentration of degradable particulate COD;

Θ = hydraulic retention time of the system (9).

Knowing the influent and effluent concentrations of degradable particulate COD and the HRT of the system, the hydrolysis rate is easily calculated.

Data given in Tables D-3 and D-4 in Appendix D were used to calculate the concentrations of COD for both feed subphases of Phases II and III. Particulate COD values were obtained by subtracting the most soluble portion of each sample, that is COD contained in the less than 0.04 micrometer size range, from the total COD of the feed sludge. The feed sludge total COD was used because essentially no stabilization

Table G-1. Data used in calculation of the hydraulic rate constants for the acidogenic reactor

Phase of Study	Total COD, mg/L	Influent Soluble COD, mg/L	Effluent Soluble COD, mg/L	Influent Particulate COD(F _o) ^a , mg/L	Effluent Particulate COD(F) ^b , mg/L	Hydraulic Rate Constant (k _h), days ⁻¹
Phase II. 20-day HRT						
Primary Sludge	42000	5336	7591	36664	34409	0.066
Homogenized Sludge	41569	5835	8559	35734	33010	0.083
Phase III. 10-day HRT						
Primary Sludge	42000	5336	8099	36664	33901	0.082
Homogenized Sludge	41569	5835	10366	35734	31203	0.145

^aInfluent Soluble COD subtracted from Total COD.

^bEffluent Soluble COD subtracted from Total COD.

occurred in the acidogenic reactor, only transformation, and therefore, the total COD of the feed sludge will correspond to the reactor contents. As previously stated, the HRT of the system was one-day throughout the study.