University of Manitoba

COMPARISON OF RESULTS OBTAINED FROM MESOPHILIC AND THERMOPHILIC ANAEROBIC SLUDGE DIGESTION

by

GERARD F. BUYDENS

A Thesis
Submitted to the
Faculty of Graduate Studies
in Partial Fulfillment
of the Requirements of
Master of Science

Department of Civil Engineering

Winnipeg, Manitoba
October, 1972



ABSTRACT

The objective of this study is the investigation of the phenomenon of thermophilic digestion and the evaluation of this phenomenon by comparison with mesophilic digestion.

The performance of these digesters is evaluated by studying the combined effects of temperature, loading, and retention time on each of the following parameters: reduction in volatile matter, gas production, gas quality, volatile acid concentration, pH level and alkalinity.

Thermophilic digestion was achieved by raising a mixture of 30 per cent digested sludge and 70 per cent raw activated sludge to a temperature of 52.8°C.

The results obtained in this study indicated that no benefits were derived from thermophilic digesters operated at comparable loading rates and retention times as mesophilic digesters.

AVKNOWLEDGEMENT

The author wishes to express his sincere appreciation to the following for their help and encouragement in the preparation of this thesis.

Dr. A.B. Sparling, Department of Civil Engineering, University of Manitoba, for his advice in carrying out and preparing this thesis.

Mr. A. Pick, Research Engineer, City of Winnipeg, for his interest and advice.

The staff at the North End Waste Treatment Plant for their advice and co-operation in carrying out the project.

TABLE OF CONTENTS

			Page
Abstract	Ξ.		iii
Acknowledgements			iv
List of	Figu	ures	vi
Symbols	and	Conversions	viii
Chapter	1.	Introduction .	1
Chapter	2.	Theory of Anaerobic Digestion	5
Chapter	3.	Literature Review	22
Chapter	4.	Objective of Study	38
Chapter	5.	Apparatus	39
Chapter	6.	Procedure	44
Chapter	7.	Experimental Results and Discussion	49
Chapter	8.	Conclusions	88
Bibliography			
Appendix			

LIST OF FIGURES

Figure	<u>1</u>	Page
1.	Methane fermentation of a complex organic waste.	6
2.	Time required for digestion.	10
3.	Conventional digester.	16
4.	High rate digester	18
5.	Anaerobic contact process.	21
6.	Average daily gas production.	26
7.	Gas production vs temperature.	27
8.	Average per cent destruction of volatile matter as a function of the temperature at which the digester is operated.	28
9.	Per cent reduction in volatile matter vs temperature.	29
10.	Experimental set-up used for thermophilic digestion.	40
11.	Experimental digester and gas collection device.	41
12.	Alkalinity and pH vs loading @ 37°C & 12 day SRT.	55
13.	Alkalinity and pH vs loading @ 52.8°C & 12 day SRT.	56
14.	Alkalinity and pH vs loading @ 52.8°C & 8 day SRT.	57
15.	% volatile solids vs time - Mesophilic digestion	62
16.	% volatile solids vs time - Thermophilic	63
17.	digestion. % volatile solids vs time - Thermophilic digestion.	64

Figure		Page
18.	% volatile solids vs time - Thermophilic digestion	65
19.	% volatile solids destroyed vs loading at 37°C.	66
20.	% volatile solids destroyed vs loading at 52.8°C	67
21.	Grams of volatile solids destroyed per day vs loading at 37°C and 12 day SRT.	70
22.	Grams of volatile solids destroyed per day vs loading at 52.8°C and 12 day SRT.	71
23.	Grams of volatile solids destroyed per day vs loading at 52.8°C and 8 day SRT.	72
24.	Gas produced vs loading at 37°C and 12 day SRT.	73
25.	Gas produced vs loading at 52.8°C and 12 day SRT	. 74
26.	Gas produced vs loading at 52.8°C and 8 day SRT.	75
27.	Volatile solids destroyed per day vs gas produced at 37°C.	77
28.	Volatile solids destroyed per day vs gas produced at 52.8°C.	78
29.	Gas produced per gram of volatile solid destroyed vs loading at 37°C.	7 9
30.	Gas produced per gram of volatile solid destroyed vs loading at 52.8°C.	81
31.	Carbon dioxide content of sludge gas vs loading at 37°C.	82
32.	Carbon dioxide content of sludge gas vs loading at 52.8°C and 12 day SRT.	83
33.	Carbon dioxide content of sludge gas vs loading at 52.8°C and 8 day SRT.	84
34.	Volatile acid concentration vs loading.	86
35.	Volatile acid concentration vs temperature.	87

SYMBOLS AND CONVERSIONS

VS - volatile solids

TS - total solids

VM - volatile matter

VA - volatile acids

°F - degrees Fahrenheit

°C - degrees Centigrade

ppm - parts per million

CH4- methane

CO₂- carbon dioxide

% - per cent

cc - cubic centimetre

gms - grams

lbs - pound

Centigrade to Fahrenheit Conversion Table

```
0^{\circ}C =
         32.0°F
                   41^{\circ}C = 105.8^{\circ}F
                                       58^{\circ}C = 136.4^{\circ}F
 5
         41.0
                   42
                         = 107.6
                                       59
                                             = 138.2
10
         50.0
                   43
                         = 109.4
                                       60
                                             = 140.0
15
         59.0
                   44
                         = 111.2
                                       61
                                             = 141.8
20
         68.0
                   45
                         = 113.0
                                       62
                                             = 143.6
25
         77.0
                   46
                         = 114.8
                                       63
                                             = 145.4
30
         76.0
                   47
                         = 116.6
                                       64
                                             = 147.2
31
         77.8
                   48
                         = 118.4
                                       65
                                             = 149.0
32
         79.6
                   49
                         = 120.2
                                       66
                                             = 150.8
33
         81.4
                   50
                         = 122.0
                                       67
                                             = 152.6
                   51
34
         83.2
                         = 123.8
                                       68
                                             = 154.4
35
         95.0
                   52
                         = 125.6
      =
                                       69
                                             = 156.2
36
         96.8
                   53
                         = 127.4
                                       70
                                             = 158.0
37
                   54
         98.6
                         = 129.2
                                       71
                                             = 159.8
38
      = 100.4
                   55
                         = 131.0
                                       72
                                             = 161.6
      = 102.2
                                             = 163.4
39
                   56
                         = 132.8
                                       73
40
      = 104.0
                   57
                         = 134.6
                                       74
                                             = 165.2
```

CHAPTER 1

INTRODUCTION

Human population has steadily increased since the start of civilization. One of the factors contributing to this is the increased life span of man due to technological advances in many parts of the world. Many authorities fear that, in time, earth will be unable to support its population if present trends in waste disposal and food production are continued.

The amount of waste produced is related to the population and therefore, it is quite evident that an increase in population implies a corresponding increase in domestic wastes. Increased industrialization brought on by a growing population has increased the amount of industrial wastes requiring stabilization. The concentration of the majority of the population into large urban areas has also added to the problem of waste disposal. The continual concentration of domestic and industrial wastes due to urbanization have resulted in waste loads which are beyond the stabilization capacity of natural processes occurring within the urban areas.

Disposal of wastes into water courses in the past

was an adequate method, since the smaller amounts of waste were diluted to such an extent that the natural processes could easily stabilize it without evidence. The growing population of the present is demanding an increased potable water supply. It is therefore essential that all possible sources of water are maintained in a state of the highest quality. This increasing demand for clean water makes it necessary that more efficient means of waste stabilization be developed in order to prevent any further pollution of water courses.

Many methods of waste treatment have been developed and used over the years. The main problem associated with most waste treatment processes has been disposal of the accumulating sludge. The problem of sludge disposal will probably become more severe as population increases, unless there are some improvements in present methods, or development of new methods of sludge disposal.

The anaerobic sludge digestion process, one of the many sludge treatment processes in use today, has many advantages over other methods of organic waste treatment, and may be the answer to the problem of sludge disposal. Some advantages of anaerobic treatment are:

- (1) a high degree of waste stabilization is possible
- (2) the volume of waste biological sludge solids produced is lower than in aerobic treatment.
- (3) anaerobic treatment has a lower nutrient require-

ment than aerobic treatment

- (4) anaerobic treatment, unlike aerobic treatment, does not require free oxygen for treatment
- (5) methane, one of its end products, is a useful by-product.

This process is widely used for stabilization of municipal waste sludges and has good potential for the treatment of many industrial wastes.

There are certain environmental conditions that favor growth in a biological system and others that will hinder it. The main environmental conditions which affect an anaerobic digestion system are temperature, pH, and available nutrients.

The effect of temperature on the anaerobic digestion process is considered to be a most complex factor.

Temperature has long been recognized as an important factor controlling the rate and course of digestion of sewage sludge. Most authorities seem to agree that the two distinct temperature ranges for anaerobic digestion are the mesophilic zone (30 to 40°C) and the thermophilic zone (49 to 57°C).

There are certain words and phrases that may cause confusion if they are not clarified. The most obvious phrase that may cause confusion is that of "retention time"

or "detention time". They are used interchangeably by authors and when used in connection with a reference in this study, they shall appear as used by the original author. Due to preference, the phrase "retention time" will be used.

There are many instances where the word "digester" appears in this study. It will be referring to anaerobic digesters in all cases.

Another phenomena that may lead to confusion is that of temperature scales. The centigrade scale will be used predominantly, but all references will include the temperature scale as used by the specific author.

A conversion scale is printed on the page of Symbols and Conversions, immediately following the Table of Contents, for the convenience of readers.

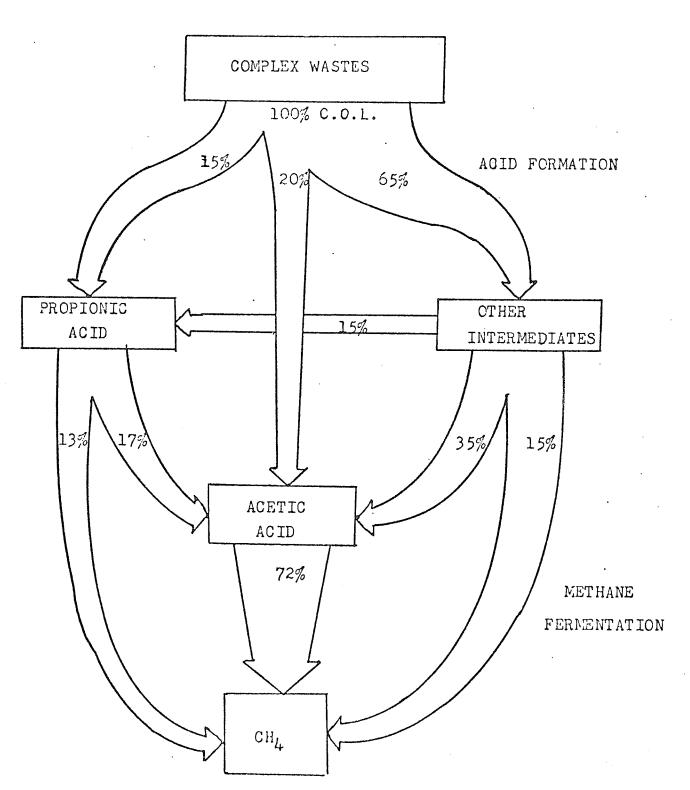
CHAPTER 2

THEORY

The process in which the organic materials are decomposed by biological action in an environment which is devoid of free oxygen is usually referred to as anaerobic digestion. This process has been widely used in sewage treatment plants. On completion of anaerobic digestion, a residue remains that is relatively stable and has had a substantial reduction in the initial organic content of the sludge. This has come about since only a small portion of the waste is converted into new cells, with the major portion of the degradable wastes being converted into methane gas and carbon dioxide [1]. The gas produced can be collected and may be used on site for heating purposes.

Anaerobic digestion is biological in nature with the decomposition being carried out in two stages by two separate groups of organisms, namely; acid forming bacteria and methane forming bacteria. Figure 1 is a schematic diagram showing the various ways the organic compounds may be broken down. In the first stage, the complex wastes are broken down by saprophytic acid formers. Saprophytic bacteria can reproduce quickly and are abundant in sewage. These

FIGURE 1



Methane fermentation of a complex organic waste. (McCarty)

bacteria are a highly mixed culture that brings about the liquefaction of organic matter by excreting extra-cellular enzymes. The degree of possible bio-degradability is dependent upon the composition of the matter. The acid forming bacteria convert the complex organics into simple short chain organics or volatile acids in a process called liquefaction. Although no waste stabilization occurs during this first stage of treatment, the organic matter is converted into a form suitable for the second stage of treatment.

Waste stabilization is achieved during the second stage of anaerobic treatment. In this stage, the organic acids or volatile acids are converted by the methane producing bacteria or methane formers into the gaseous end products (carbon dioxide and methane gas) in a process referred to as gasification.

The methane bacteria are strictly anaerobic and even trace amounts of oxygen may prove fatal [1,2,3]. These bacteria are also extremely sensitive to temperature and pH variations. There are several groups of methane formers, each of which is capable of fermenting a particular type of organic compound. Methane forming organisms require carbon dioxide for the reduction of volatile acids to methane. This conversion is accomplished by intracellular enzymes secreted by the bacteria. In the chemical reaction, carbon dioxide acts as the hydrogen acceptor and is reduced

to methane gas [1,2,3]. Several different types of methane bacteria are required in a complex system such as anaerobic sludge digestion.

Methane bacteria are well known for their slow reproductive rate. The slow growth and low rate of acid utilization normally represents the limiting step, around which the anaerobic treatment process must be designed. The methane bacteria that live on acetic and propionic acid grow quite slowly and sludge retention times of four days or longer are required for the growth of the culture [1,3].

The complete process of gasification involves various different groups of methane formers and a period of weeks is usually required before a balance of all the required bacterial cultures is achieved. An acclimation period of a few weeks is required when starting up a digester to ensure that adequate bacteria cultures have developed.

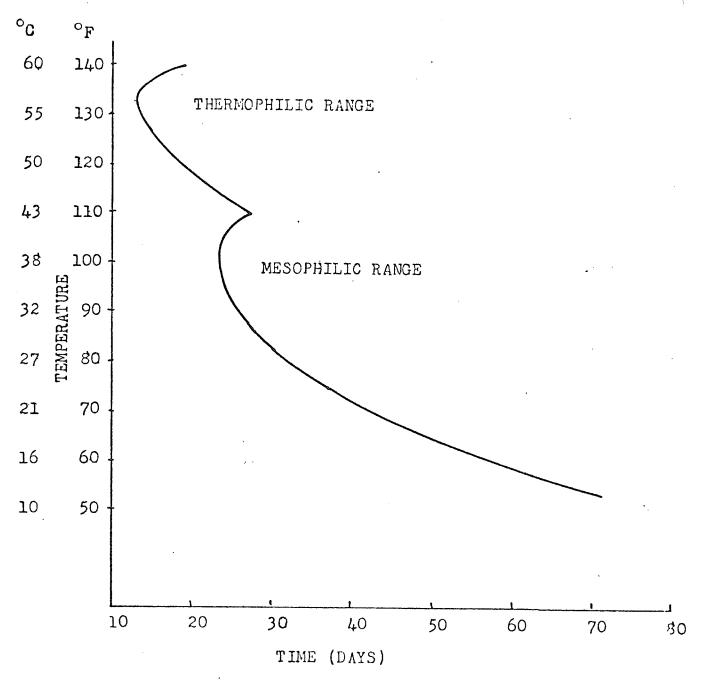
While there are many different methane forming bacteria, there are also many different acid forming bacteria. A balance among these organisms is required in order to obtain good waste stabilization. The test for the concentration of volatile acids is used as an indicator of the degree to which this balance has been attained [1,2,4,5]. The methane bacteria utilize the acid intermediates as rapidly as they are formed in a balanced system. If liquefaction proceeds too quickly, or gasification too slowly, an acid build-up results. A build up of volatile

acids usually results when the methane formers have not been able to utilize the volatile acids as quickly as they formed, and a large build up of volatile acids lowers the pH of the media which, in turn, inhibits the methane formers. This situation is usually caused by a variation of one or more environmental parameters or the introduction of a toxic substance into the digester. The gasification stage will stop if this situation is not corrected. The result is referred to as a stuck or sour digester.

Anaerobic decomposition is greatly affected by environmental conditions [1]. Environmental conditions that may create problems if not maintained constant are temperature, pH, and feed rate. A sudden change in any of these may result in an upset digester. Other environmental conditions that require attention are the amount of available nutrients and the amount of toxic material present in the feed sludge.

Temperature is one of the most important factors affecting the performance of an anaerobic digester. The rate of gas production is directly related to the temperature. An increase in temperature reduces the retention time required for digestion, as shown in Figure 2. Temperature, although it has no effect on the sequential mechanism of anaerobic sludge digestion, does affect the duration of each reaction and thus the time required for completion of the digestion process [1,2,6]. The final degree of

FIGURE 2



Time required for digestion. (Fair and Moore)

waste stabilization is the same, despite the slower rates of digestion at lower temperatures, if the organic matter is detained long enough.

There are two significant temperature zones, mesophilic and thermophilic, in which appreciable anaerobic sludge digestion may occur. Lower temperature zones do exist in which anaerobic digestion may occur; the temperate zone, between 10 to 28°C and possibly, a cryophilic zone, below 10°C [6].

Thermophilic organisms are responsible for digestion in the highest temperature zone, above 42°C (107.6°F). The mesophilic zone is a zone of moderate temperature, existing between the temperatures of 28 to 42°C (82.4 to 107.6°F), in which mesophilic organisms are predominant. These distinct temperature zones were found by Fair and Moore [6] during their study (Figure 2).

The temperature of a digester, once established, must remain constant if the most efficient digestion process is to occur. The balanced bacterial population required for optimum digestion will only exist if the prescribed temperature is maintained. Variations of only a few degrees can cause serious digester upset due to the inhibition of the methane formers [1,2,3,8].

There are various means of feeding a digester. The oldest method is that of batch feeding, a procedure in which the system is loaded once, sealed and allowed to

proceed until the digestion is completed. The method of periodic feeding and sludge withdrawal is usually used in small installations, or in pilot plants. The volume of digested sludge withdrawn in this process is usually equal to the volume of raw sludge fed. The more recent and most popular method is that of continuous feed and withdrawal. Equal volumes of raw and digested sludge are fed and withdrawn continuously in this process. method is used in larger plants where the feed load is fairly constant. This method enjoys added popularity, because it does away with the build-up of volatile acids that occur in systems using periodic loading due to the sudden variation of available raw sludge. The process becomes adjusted to a continuous incoming load and the microbial population is maintained in a state of equilibrium.

A sludge concentration of between ten and twelve per cent solids appears to be the maximum allowable to ensure proper digestion. This may vary with the volatile content of the raw sludge [1,5]. The allowable loading rates vary according to the type of digester employed. Loadings as low as 0.1 pounds of volatile solids per cubic foot of digester per day (lbs. VS/cu. ft./day) to as high as 0.4 lbs. VS/cu. ft./day have been successfully used [1,2,5,6,7].

The system must maintain anaerobic conditions as

even a small quantity of oxygen will be quite detrimental to the methane formers and other anaerobic organisms.

This is another of the important environmental conditions.

Sufficient biological nutrients must be available for optimum growth [1,2]. These nutrients are nitrogen, phosphorous, and other materials in trace amounts. These nutrients are normally present in municipal wastes, but industrial wastes may not contain all the necessary nutrients as they are usually more specific in composition. Nutrients may then have to be added to industrial wastes if optimum digestion is to take place.

Another of the more important factors affecting anaerobic sludge digestion is pH. pH is used as an indication of how well the anaerobic digestion process is proceeding. Anaerobic digestion can proceed quite well within a pH range of 6.6 to 7.6, but the optimum pH range is 7.0 to 7.3 [1,2,3,5]. Anaerobic digestion can function outside of these pH ranges but it is severely inhibited. The prevailing acidic conditions at a pH level below 6.2 can be quite toxic to the methane bacteria.

The concentration of toxic matter present in the feed sludge is an important consideration which may affect the anaerobic digestion process, and it should be kept as low as possible to ensure optimum anaerobic digestion. The most common of these toxic materials are the heavy metals, sulfides, ammonia, alkali and alkaline earth salts. The

degree of toxicity is dependent on the material present and its concentration [1,2].

One of the conveniences of anaerobic digestion is that size and shape of the digester do not affect digestion rates. Laboratory studies can therefore be conducted to evaluate digester performance at various temperatures, loading rates and solids concentrations without much concern for scale effects.

Gas production is an indication of the degree of waste stabilization being achieved. An anaerobic sludge digester is considered biologically balanced if its gas production exceeds 8 cubic feet per pound of volatile solids fed per day. The methane content of the sludge gas is approximately 65 per cent when good digestion is occurring. The remaining components are carbon dioxide, making up approximately 30 per cent, hydrogen sulfide, oxygen, nitrogen and hydrogen as a group making up the other five per cent.

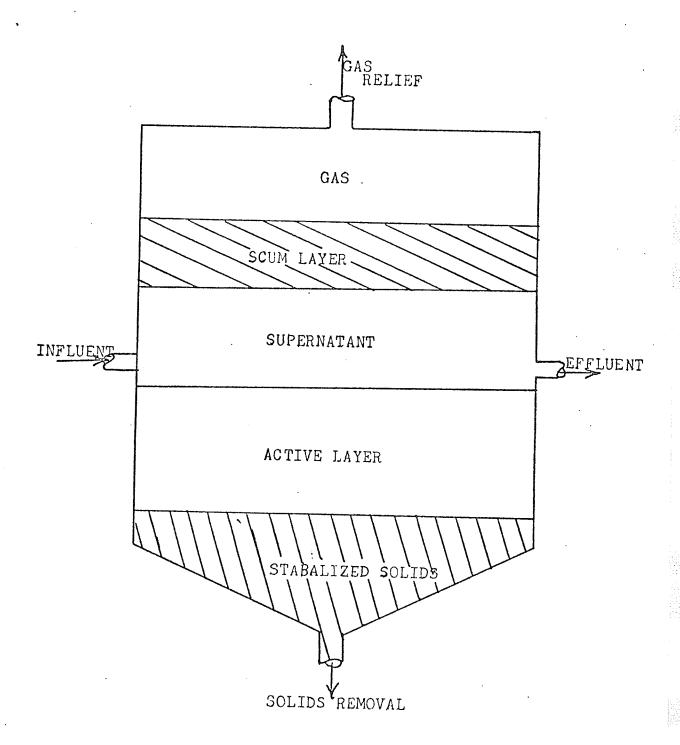
There are essentially three types of digestion processes used today: the conventional, high rate, and anaerobic contact process. The conventional digester, the oldest of these processes, was used as early as 1890. The high rate digester, a modification of the conventional digester, was developed next during the 1930's. The anaerobic contact process is the most recent and became quite popular for treating dilute wastes in the late fifties.

A conventional digester consists of a digestion tank containing waste and bacteria responsible for anaerobic decomposition. There is no mixing in conventional digesters and stratification occurs as shown in Figure 3. Conventional digesters may or may not be heated. Heating decreases the detention time which normally ranges from 30 to 60 days. Maximum loading rates which were used successfully are about 0.03 pounds of volatile solids per cubic foot of digester per day [1,2,5]. The conventional digester employs intermittent feeding and sludge withdrawals, usually of The gas produced rises above the liquid equal volume. wastes as the wastes are stabilized and the stabilized solids settle to the bottom of the tank. The sludge gas may be tapped off the top of the tank while stabilized solids are pumped out the bottom, as shown in Figure 3. Raw sewage is usually added from the top.

The physical size of the tank required because of the long retention time, the low loading rate and thick scum layer formation is one of the main disadvantages of a conventional digester [2,5]. The digestion process is carried out in about one-third of the volume of the tank, with the remaining two-thirds being taken up in the storing of the stabilized solids, separated liquid and a scum layer.

The high rate anaerobic digester is a modification of the conventional digester. High rate digestion is a much more efficient process due to the constant mixing

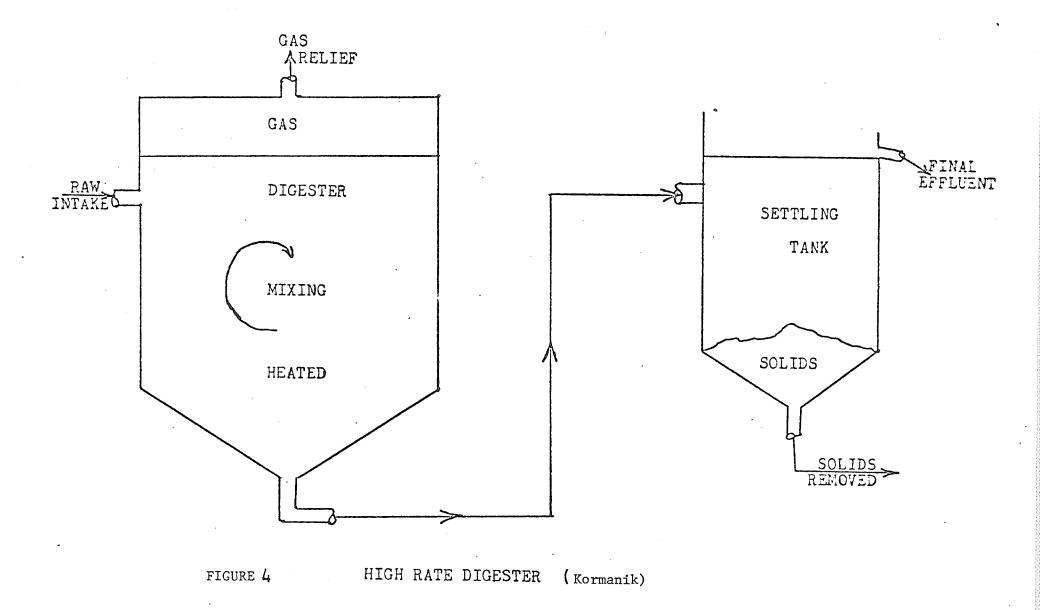
FIGURE 3



CONVENTIONAL DIGESTER (Kormanik)

which creates the greatest possible contact between bacteria and raw sludge. A high rate digester is usually operated at an optimum temperature so that maximum utilization of substrate by the bacteria is possible. This anaerobic system employs continuous feeding of raw sludge and withdrawal of equal quantities of stabilized sludge. digester contents are thoroughly mixed by use of either mechanical means and/or the recirculation of sludge gas through the digesters. The high rate digester has a detention time of 10 to 15 days. The loading rates vary from 0.1 to 0.4 pounds of volatile solids per cubic foot of digester per day [1,2,5]. The sludge gases rise above the sludge and collect in the space above the liquid in The stabilized sludges are pumped off the this process. bottom of the digester and are usually sent to a holding tank where final settling occurs. The settled solids portion of the digested sludge is pumped out to drying beds, vacuum filters or spread directly onto agricultural land. The liquid portion of the digested sludge has a high biological oxygen demand and must be returned to the treatment plant for further treatment. There is no noticeable occurrence of stratification or scum layer in a high rate digester.

A schematic diagram of a typical high rate digester is shown in Figure 4. The high rate digester requires a much smaller tank than the conventional digester since the



whole digestion tank is being used for digestion and its retention time is much shorter.

The anaerobic contact process is the most recent development in the field of anaerobic digestion. Its main advantage, over the other types of anaerobic digesters, is its ability to economically treat dilute organic wastes [1]. Anaerobic contact digesters are considered to be high speed digesters which can be operated at detention times as low as three to four hours with loading rates of about 0.1 to 0.2 pounds of volatile solids per cubic foot of digester per day [1,2].

The physical make-up of an anaerobic contact digester is much the same as that of a high rate digester. The major difference between them is the recycling of settled solids in the anaerobic contact process. The solids are recycled back to the digester where they are mixed with the raw substrate as it enters the digester. The recycling rate may be as high as 150 per cent of the raw flow. The recycling process is used to seed the incoming substrate. Since the organisms are continually being recycled the digester is not dependent on population build-up. This process, like the high rate digester entails continuous feeding, withdrawing and mixing.

Numerous research projects have been conducted in an attempt to find a method of improving the rate of settleability of the digested sludge. Of these, the vacuum

degasification process has proved the most effective [2]. The vacuum degasification device, if placed between the digester and the settling tank, can remove the suspended gases which tend to keep the solids in suspension, improving the rate of settleability greatly.

These digesters require an even smaller volume than the others because of the short detention time required.

A typical anaerobic contact process is shown in Figure 5.

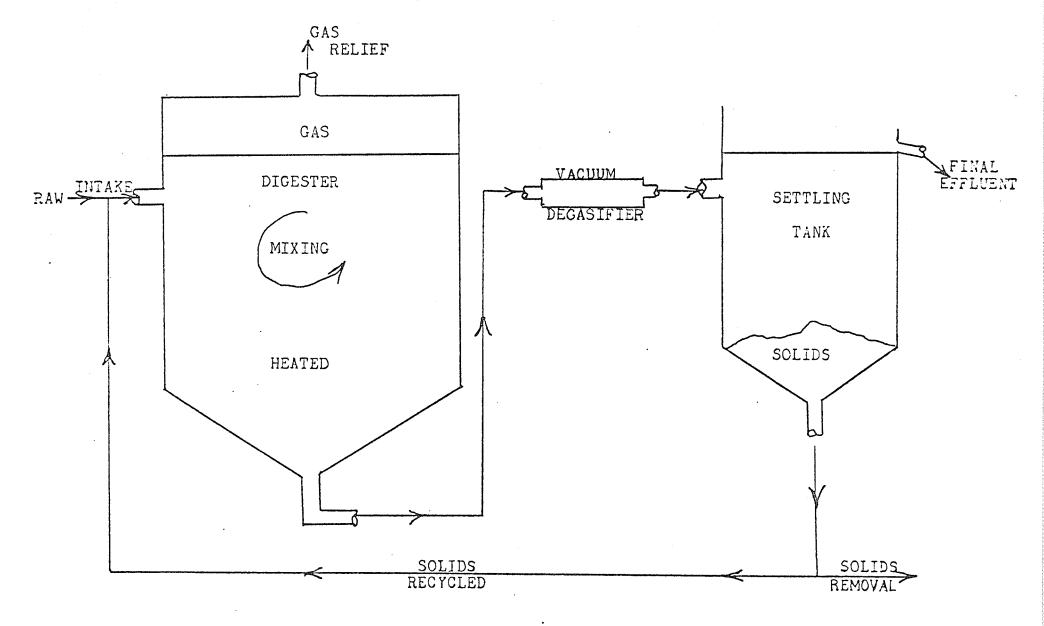


Figure 5 ANAEROBIC CONTACT PROCESS (KORMANIK)

CHAPTER 3

LITERATURE REVIEW

Anaerobic sludge digestion has become widely adopted although relatively little is known about the metabolic processes that occur and the existing relationships between the many factors that effect its efficiency. A knowledge of specific conditions required for this process is indispensable when contemplating the design of digestion tanks and effective control of their operation. One of the fundamentals, studied by use of a batch fermentation, is the relationship between the rate and degree of organic matter digested in sludge and the temperature at which digestion occurred.

The influence of temperature on sludge digestion has been studied by many authors. The familiar mesophilic-thermophilic curve (Figure 2) reported in many publications was published by Fair and Moore [6] in 1934. Heukelekian [9], as early as 1933, had noticed that anaerobic decomposition seemed to prosper in certain temperature zones. The importance of this parameter, temperature, is indicated by the number of researchers who have studied it [6,7,9,10, 11,12,13]. There is some difference of opinion but the

optimum temperature for mesophilic digestion is assumed to be somewhere in the range between 30 to 38°C (85 to 100°F), whereas, that for thermophilic is between 49 to 60°C (120 to 140°F). The range between 38 to 49°C (100 to 120°F) is considered to be one in which neither the mesophilic nor thermophilic organisms can prosper [4,6,9,10,11,13]. Golueke [12] did not find this inhibitory zone but found that digestion occurred equally well at all temperatures from 35 to 60°C, once a suitable population had built up. He did indicate that at 45°C the ratio of methane to carbon dioxide content of the sludge gas was low. Garber [7] in his studies at Los Angeles also failed to detect this transition zone.

The optimum mesophilic and thermophilic temperature or temperature zone as reported by each researcher was:

Fair and Moore [6]; 33°C for the mesophiles and 55°C for the thermophiles, Heukelekian [9]; 28°C for the mesophiles and the range 50 to 55°C for the thermophiles. Both Golueke [12] and Garber [7] failed to find this transition zone, but Golueke [12] found that a rapid increase in volatile solids reduction occurred between 30 and 35°C which seems to imply that the optimum for most members of the mesophiles exists at 35°C. He also found that once a suitable population had built up, digestion proceeded equally well at all temperatures between 35 to 60°C, provided the set temperature was maintained. Rowe [11]

in his study operated digesters at 35°C in the mesophilic range and at 55°C for the thermophiles. He considered both temperatures optimum for their respective ranges. Malina [10] operated digesters simultaneously at temperatures of 32.5°C, 42.5°C, and 52.5°C. He found that digestion proceeded quite well at 32.5°C and at 52.5°C but found that digestion was inhibited at 42.5°C. Malina found that the rate of gas production was a minimum at a temperature of 42.5°C and that this effect was more pronounced at a loading rate of 0.3 pounds of volatile solids per cubic foot of digester per day than at a loading of 0.1 pounds of volatile solids per cubic foot per day. Pohland and Bloodgood [4] found that digestion proceeded quite well at 37°C but it was inhibited at both 52°C and at 60°C. They found that gas production was low, volatile acids were high and that the methane to carbon dioxide ratio was low in the thermophilic range. Bloodgood [20] indicated that he had not been successful in establishing the phenomenon of thermophilic digestion and after trying for about ten years was beginning to doubt if it actually existed. The literature review makes one aware of the conflicting results obtained by various researchers.

Golueke [12] and Malina [10] have carried out studies that can be paralleled to this study and it would be beneficial to draw further comparisons of their results. Noticeable differences were apparent on comparing the

findings of Golueke and Malina. These differences were noticed, as shown in Figures 6 and 7, when their findings on gas production per day at various temperatures were compared. Figure 6 is a graph of Golueke's findings, while Figure 7 represents Malina's results. The curves, Figures 8 and 9, representing the relationship between per cent destruction of volatile matter and temperature, established respectively by Golueke and Malina, do not show much resemblance. One thing that must be kept in mind when comparing the results of these two studies is that Golueke carried out his work using a retention time of 30 days and a loading rate of 0.09 pounds of volatile solids per cubic foot of digester per day while Malina used retention times of 12 and 6 days with loading rates of 0.1, 0.2 and 0.3 pounds of volatile solids per cubic foot of digester per day. These research projects were conducted in different cities and therefore the sludge characteristics could be different and consequently could have affected their respective results. The parameters within which these different researchers worked must also be kept in mind when comparing their results with those of others.

The teams of Fair and Moore [6], Maly and Fadrius [13] and Heukelekian [9] used conventional batch fed digesters for their studies. Malina [10], Golueke [12], Hill [14], Torpey [15] and Pohland and Bloodgood [4] used

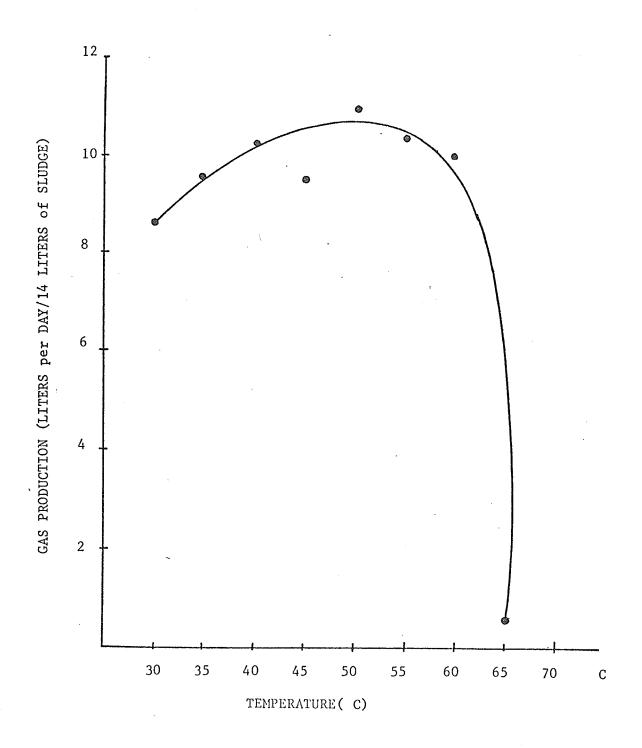


FIGURE 6 AVERAGE DAILY GAS PRODUCTION (Golueke)

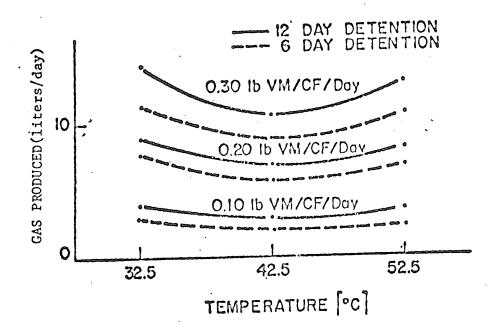


Figure 7 GAS PRODUCTION vs TEMPERATURE (Malina)

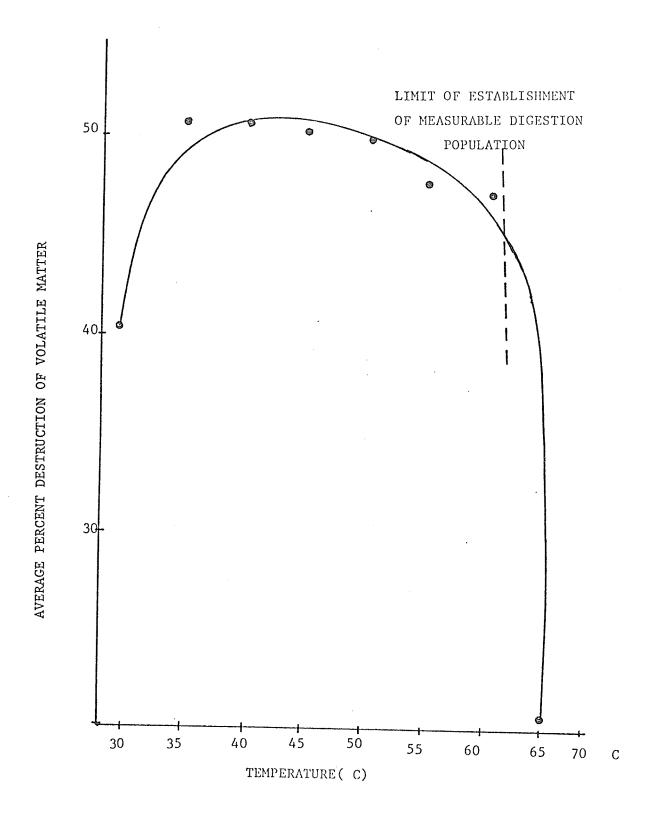


FIGURE 8 AVERAGE PERCENT DESTRUCTION OF VOLATILE MATTER AS A FUNCTION OF THE TEMPERATURE AT WHICH THE DIGESTER IS OPERATED (Golueke)

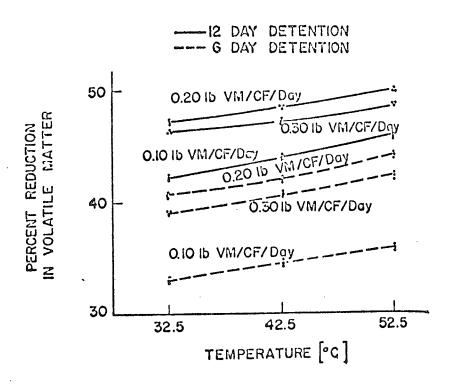


Figure 9 PERCENT REDUCTION IN VOLATILE MATTER
vs TEMPERATURE (Malina)

high rate anaerobic digesters for their respective studies.

Solids retention time is an important parameter governing the efficiency and operation of all anaerobic digestion processes although it does have a slightly different meaning when used in reference to batch fed or continuously fed digesters.

Few researchers agree on the method of arriving at a value for digestion time and according to Fair and Moore [6] there is no one time of digestion for a given temperature. The rate at which digestion proceeds is controlled by many variables, chief of which is the nature of the material to be digested.

Some researchers have defined detention time as the time required to produce 90 per cent of the total gas. They consider the digestion process complete when gas production drops to 10 cubic centimetres per gram of volatile matter [6,9]. Maly and Fadrius [13] considered the process complete when gas production ceased or was reduced to a negligible rate. They calculated digestion time as the time interval during which intensive gas production occurred.

Detention times of 30 to 60 days are required for batch fed digesters, although cases have been cited where digestion time was shorter. Fair and Moore [6] found that digestion time for a given substrate varied according to the temperature, as shown in Figure 2.

The detention time in a continuously fed digester is usually referred to as a theoretical detention time. Solids retention time or theoretical solids retention time as given by McCarty [1] is the total weight of suspended solids in the anaerobic digester system divided by the total weight of suspended solids leaving the system per day, including both that deliberately wasted and that passed out with the plant effluent. Solids retention time is dependent upon several variables, the main ones being temperature at which digestion takes place, loading rate and the degree of stabilization desired.

The limiting detention time for complete anaerobic digestion is the rate at which the methane formers reproduce [1,3]. Detention time is an independent factor in high rate and conventional continuously fed digesters while it is a dependent factor in a conventional batch fed digester. Detention times required in a conventional continuously fed digester vary from 30 to 60 days [1,2,5]. Detention times of 15 to 30 days are required in a high rate digester employing thorough mixing, although shorter times have been used successfully [1,2,4,5,10,11].

Malina [10] used detention times as short as six days with good results. Torpey [15] in a pilot plant study showed that a high rate digester can be maintained in a condition of stable operation at detention times as short as 3.2 days.

There is a relationship between solids retention

time, temperature, loading rate and stabilization achieved. This fact was noted by researchers during their work on high rate anaerobic digesters.

Golueke [12] found that good results could be obtained at a loading rate of 0.09 pounds of volatile solids per cubic foot of digester per day and a detention time of 30 days at all temperatures from 35°C to 60°C. Golueke [12] found that the destruction of volatile solids under these conditions varied between 48 per cent at the high temperatures and 52 per cent at the low temperatures. Malina [10] found that he could obtain good results using retention times of six and twelve days at temperatures of 32.5°C and 52.5°C and loading rates of 0.1, 0.2 and 0.3 pounds of volatile solids per cubic foot of digester per day. Malina [10] found that gas production was greater at the twelve day detention time than at the six day detention time for comparable temperatures and loading rates. volatile matter destroyed was found to be greater at the twelve day detention time for all cases. Malina [10] concluded that reduction in volatile material increased with increase in temperature, loading rate and detention time. He also concluded that the loading rate and detention time have a significant influence on the digester performance. The effect of loading was found to be dependent on the detention time. Temperature also had a significant influence on digester performance, but the effect of

temperature is independent of loading rate and detention time [10].

Pohland and Bloodgood [4] in their pilot plant studies operated digesters at temperatures of 97, 126 and 140°F (36, 52.5 and 60°C). They started with a loading rate of 0.037 pounds of volatile solids per cubic foot of digester per day and gradually increased the loading rate intending to eventually induce failure. In this manner they would be able to get data from both normal and retarded digesters. They found that digestion proceeded quite well at 97°F, but that the results at 126°F and at 140°F indicated retarded digestion. It was noted at temperatures of 126°F and 140°F that the volatile acids concentration was high, gas production was low and the methane to carbon dioxide ratio was low. They noticed in their study that as the volatile acids concentration increased, a corresponding decrease in gas production occurred. This indicated that the liquefaction of organic wastes to volatile acids was functioning but that gasification of volatile acids to gas was inhibited. This thereby indicated the sensitive nature of the gasification process to adverse environmental changes. The results obtained from anaerobic sludge digesters operated at temperatures of 97, 126 and 140°F under the influence of increased loadings indicated that the equilibrium between the various digestion indices such as volatile solids, total alkalinity, total and ammonia nitrogen, total volatile acids, pH, gas production and carbon dioxide content of the gas may have been so disturbed as to result in inhibition of the entire process and the development of conditions ascribed to retarded and severely retarded digestion.

Torpey [15] in his studies on a pilot plant high rate digester found that he was able to use a loading rate of 1.15 pounds of solids per cubic foot of digester per day at retention times as low as 3.2 days. The pilot plant was equipped so that the temperature could vary between 90 and 100°F. Torpey [15] was able to successfully operate a digester at a retention time of 3.2 days, but he did indicate that the gas production per pound of volatile solids fed was lower at this detention time than at one of 14 days.

Hill and Schroeder [14] set up a pilot plant at a temperature of 35°C initially, and a retention time of 20 days. After completion of one retention period, the temperature was increased 1.0°C per day until temperatures of 38, 41 and 44°C had been attained for three different groups of digesters. All digesters were operated for one retention period once these temperatures were attained and the results were recorded. Hill and Schroeder [14] found gas production was the greatest at 38°C. After one retention period at these temperatures, the temperatures were again increased until temperatures of 42.5, 46 and

52°C were attained. Gas production was negligible in all digesters at these temperatures. Gas production ceased immediately as the digester temperature was raised above 41°C. Hill and Schroeder [14] noted a large increase in volatile acid concentration at 42.5°C and that volatile acid concentration became greater as the temperature was increased above 42.5°C. They found that raising the temperature to 52°C, which is reported to be the optimum temperature for thermophilic methane forming bacteria, did not cause resumption of gas production but instead further lowered the digestion rate.

Rudolfs and Heukelekian [16] also found they were unable to achieve thermophilic digestion by using sludge produced at a mesophilic temperature and raising its temperature. They obtained satisfactory results at the thermophilic temperatures provided the sludge was seeded with a sludge produced under thermophilic conditions.

Shindala and Byrne [19] set out to operate digesters at temperatures of 105, 120, 130 and 140°F. The solids concentration of the raw sludge was also varied from 5 to 15 per cent during these tests. Sludge as thick as 15 per cent solids was successfully digested at 105°F once acclimation had taken place. The Water Pollution Control Federation Manual of Practice No. 16 [5] recommends a maximum solids concentration of 12 per cent but preferably not greater than 8 per cent. The data gathered during

their investigation indicated a severe deterioration in sludge digestibility at thermophilic temperatures as evidenced by extremely low gas yield, high volatile acid concentration and high pH.

Shindala and Byrne [17] attempted to try and achieve thermophilic digestion during the second phase of their investigation using sludge digested in the mesophilic They used ripe mesophilic sludge and gradually increased its temperature in this way hoping to achieve thermophilic digestion. Complete failure was declared after 12 days at a thermophilic temperature when it was realized that the accumulative gas production for none of the digesters exceeded 600 millilitres of gas per litre of sludge. They concluded that temperature definitely affected the digestion of a thickened sludge. They also found that deterioration in digestibility occurred for all sludges at temperatures higher than 105°F. Alkalinity and volatile acid concentrations were found to increase as digestion temperatures increased.

Summarizing the literature reviewed it becomes apparent that although similar procedures had been employed, final results were often quite different.

A summary of the more noteworthy facts found regarding the process is as follows. Fair and Moore [6] concluded that the rate at which digestion proceeds is controlled by many variables, the chief ones being the

nature of the seed material and the nature of the material to be digested. Differences in results are quite evident on comparing the results obtained from the studies conducted by Pohland and Bloodgood [4], Golueke [12] and Malina [10]. Their final results were quite different although their research was carried out within similar parameters.

Malina [10] successfully operated digesters in both mesophilic and thermophilic temperature ranges. Golueke [12] did likewise, although his results varied considerably from those obtained by Malina [10]. Pohland and Bloodgood [4] on the other hand, although able to operate mesophilic digesters, did have some trouble maintaining proper digestion in this temperature range during the course of the study. They were unable to achieve proper digestion in the thermophilic temperature range.

In all cases domestic sewage sludge from the local plant was used, but this is no assurance that it was of similar composition. It is quite likely that the difference in chemical make-up of the raw sludge may have affected the operations of their respective digesters.

CHAPTER 4

OBJECTIVE OF STUDY

The objective of this study was the investigation of the phenomenon of thermophilic digestion and the evaluation of this phenomenon by comparison with mesophilic digestion.

The performance of these digesters were evaluated by conducting studies on the combined effects of temperature, loading and retention time on each of the following parameters: reduction in volatile matter, gas production, gas quality, volatile acid concentration, pH and alkalinity.

The anaerobic digesters were operated at 37°C in the mesophilic range and at 52.8°C in the thermophilic range. These digesters were operated simultaneously within specified parameters and in such a way as to simulate high rate digesters. The digesters were operated at retention times of eight and twelve days and loading rates of 0.1, 0.2, and 0.3 pounds of volatile solids per cubic foot of digester per day. They were fed once a day and shaken manually twice a day to ensure thorough mixing. Volumes of digested sludge equal to the amount fed were withdrawn each day and analysed. The results were noted along with any other pertinent information.

CHAPTER 5

APPARATUS

The apparatus used in this study on high rate anaerobic digestion was:

- (1) constant temperature room
- (2) insulated water bath
- (3) Thermomix II water bath heater and circulator
- (4) one gallon glass jugs
- (5) graduated glass beakers
- (6) a Radiometer pH meter model 29
- (7) an Orsat Flue Gas Analyzer

The constant temperature room which housed the mesophilic digesters was maintained at a temperature of 37°C. The thermophilic digesters were immersed in an insulated water bath whose temperature was maintained at 52.8 ± 0.1°C by a water bath heater and circulator. Digesters consisted of one gallon glass jugs fitted with an outlet valve at their base. The gas collection devices were made up using one gallon glass jugs. Figure 11 is a photograph of a digester and its gas collection device. A Radiometer pH meter was used to check the pH level of the media. An

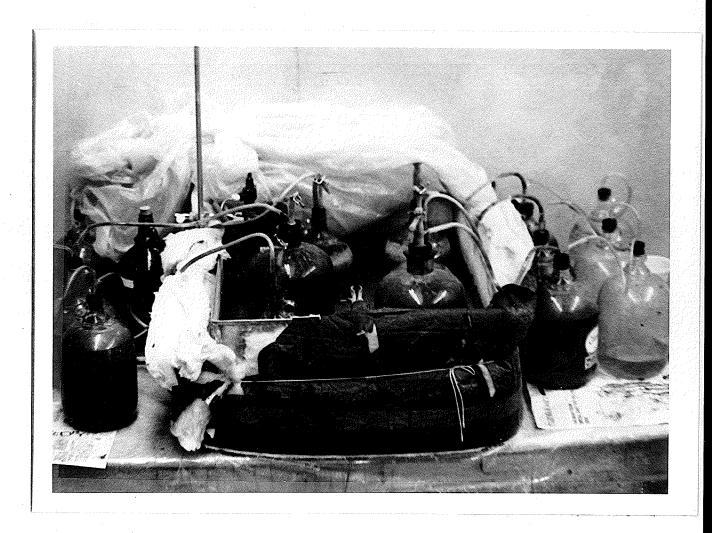


FIGURE 10 EXPERIMENTAL SET UP USED FOR
THERMOPHILIC DIGESTION

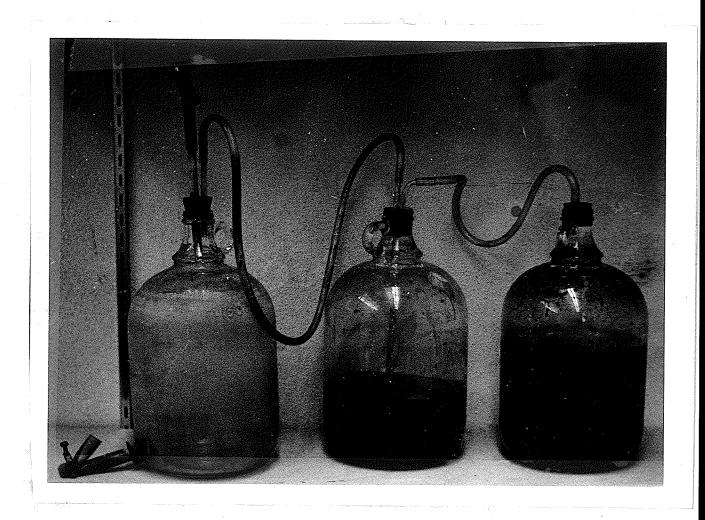


FIGURE 11 EXPERIMENTAL DIGESTER AND GAS

COLLECTION DEVICE

Orsat Flue Gas Analyzer was used to evaluate the carbon dioxide content of the sludge gas.

The operation of an anaerobic digester is not affected by either the size or shape of the container, thus, rounded gallon jugs were used. Glass jugs were used to eliminate any possible effects due to chemical or biological reactions with the container walls. One gallon glass containers were selected as they were readily available, made of an inert substance and also permitted visual inspection of the digesters.

The jugs used as digesters had a hole drilled near the base of the jug which was fitted with a plug and valve for use as the sludge withdrawal outlet. The tops were fitted with two-holed stoppers through which two glass tubes were inserted. One glass tube was used as a feed inlet and the other as the sludge gas outlet. The gas outlet was connected to the gas collection device by plastic tubing as shown in Figure 11.

The sludge gas was collected by the liquid displacement technique. The gas collection device consisted of two one-gallon glass jugs fitted with two-holed stoppers and interconnected as shown in Figure 11. To prevent any gas produced from dissolving in the liquid, a saline solution with a pH level below 4.3 was used in the gas collection device. The saline solution used, contained 200 grams of sodium sulphate and 50 grams of concentrated

sulfuric acid per 800 millilitres of water. As it was produced, the sludge gas displaced the saline solution from the first jug of the gas collection device. The displaced solution flowed through the plastic tubing into the second jug which served as a liquid reservoir. All jugs were calibrated so that the volume of liquid displaced could be read directly.

CHAPTER 6

PROCEDURE

The experimental system consisted of two mesophilic digesters and six thermophilic digesters. Digesters were operated in pairs and in this manner data was obtained in duplicate.

The mesophilic digesters were operated at 37°C, which appeared to be the optimum mesophilic temperature for digesting domestic sewage sludge. The digesters were placed in a constant temperature room which was thermostatically controlled.

The thermophilic digesters were operated at 52.8°C, midway between the quoted optimum thermophilic temperatures of 52.5 and 53°C. These digesters were immersed in a water bath which was thermostatically controlled to within ±0.1°C.

The digesters consisted of gallon jugs fitted with the required number of outlets. The total volume of all digesters was approximately 4.6 litres but they contained only 3.6 litres of digesting sludge. The difference in volume between total volume and sludge volume of the digester was used to aid mixing of the digester and also

served as a space for collecting the evolving sludge gases.

Manual shaking at regular intervals ensured thorough
mixing of the digesters.

Digesters were attended daily as soon as it appeared that a suitable culture had developed which consisted of feeding, withdrawal and resetting of gas collection device.

Digesters were fed from the same batch of raw feed to rule out possible effects due to change in raw sludge quality. A volume of digested sludge, equal to that fed, was withdrawn every day. The raw feed sludge was added to the digester through a glass tube on the top of the digester and the digested sludge was withdrawn through an outlet on the base of the digester as shown in Figure 11.

Digesters were operated for a time period sufficient to provide at least one complete displacement of the digester contents before testing was commenced. Runs in this study lasted about 25 days with 10 days being required to ensure acclimatization and tests were conducted for the next 15 days to check digesters parameters. Gas production indicated ten days was adequate for acclimatization. The consistency of results obtained from tests run during the next 15 days indicated digesters had to be acclimatized.

Mesophilic digesters were operated at loading rates of 0.1, 0.2, and 0.3 pounds of volatile matter per cubic

foot of digester per day* at a solids retention time (SRT) of 12 days.

In each thermophilic run there were six units, two at each of the three different loading rates. Loading rates of 0.1, 0.2 and 0.3 pounds of volatile solids per cubic feet of digester per day were used in the thermophilic digesters with SRT's of 12 and 8 days.

Using the procedures as outlined in Standard Methods for the Examination of Water and Wastewater [18] the total and volatile solids content of the raw sludge were evaluated in terms of gram per litre (gm/l) of raw sludge. The volume of raw sludge to be fed to achieve the different loading rates could be calculated, knowing the concentration of volatile solids.

Distilled water was added to the volume of raw sludge to make up the required volume as the volume of raw sludge required to satisfy the organic loading did not always satisfy the volume required for the set retention time. A volume of 300 millilitres was required for an SRT of 12 days while a volume of 450 millilitres was required for an SRT of 8 days.

Enough raw sludge was collected for feeding purposes for the complete run in all cases. This eliminated any

^{* 1.32} grams of volatile solids per litre = 0.1 pounds of volatile solids per cubic feet.

variation in raw sludge characteristics. The feed sludge was refrigerated, in order to prevent any biological reaction from occurring prior to feeding. The feed container was well shaken prior to withdrawing to ensure the feed was thoroughly mixed as the solids tended to settle out on standing.

Any reaction due to temperature difference between raw feed sludge and digester contents was overcome by adding heated distilled water to the raw sewage when making up the required volume. Placing these feed samples in the water bath along with the digesters in the case of thermophilic digestion or in the constant temperature room in the case of mesophilic digestion for a few hours prior to feeding, resulted in a temperature very close to that of the digester.

Tests as outlined in Standard Methods for the Examination of Water and Wastewater [18] were conducted on the withdrawn sludge after acclimatization to determine its pH, alkalinity, total and volatile solids. The volatile acid concentrations were checked using the procedure outlined in Hach D R Colorimeter Methods Manual [22].

The volume of sludge gas produced was recorded daily. The carbon dioxide content of the sludge gas produced was determined using an Orsat gas analyzer. Trace amounts of nitrogen, hydrogen, hydrogen sulfide and oxygen were present but their combined volumes made up less than five per cent

of the total volume of sludge gas [21]. The methane content of the sludge gas was determined by subtracting the carbon dioxide, nitrogen, hydrogen, hydrogen sulfide and oxygen content from the total volume of the sludge gas.

CHAPTER 7

EXPERIMENTAL RESULTS

Mesophilic digestion was easily established in experimental anaerobic sludge digesters. Seed sludge was obtained from the local waste treatment plant and placed in the experimental digesters. The sludge digesters at the local waste treatment plant are operated within the mesophilic temperature range. Digestion in the pilot plant was proceeding normally within two days and daily maintenance was commenced. Tests were performed on the digested sludge, after a suitable acclimatization period and the results were recorded.

The thermophilic anaerobic digestion process was not so easily achieved. After three unsuccessful attempts, thermophilic digestion was finally achieved. It was not possible to achieve thermophilic digestion by taking digested sludge from a mesophilic digester and raising its temperature to that desired within the thermophilic range.

The first trial involved increasing the temperature from 37°C in the mesophilic range to 53°C in the thermophilic range. Sludge taken from the local waste treatment plant was placed in experimental digesters and operated at

37°C for approximately four days. The temperature was increased to that desired within the thermophilic range once satisfactory normal mesophilic digestion had been attained. Gas production ceased almost immediately when the temperature reached 53°C. When no apparent reaction was evident after 12 days, the digester contents were discarded.

The substantial increase in temperature was suspected of being responsible for the failure in the first trial, so in the second trial, the temperature was raised in steps of 2°C per day. The temperature was increased slowly anticipating the organisms would adjust to the new temperature. Starting at 37°C, the temperature was raised in steps of 2°C per day and at this rate the desired temperature of 53°C would have been achieved within eight days. Digestion was evident during this period up to and including a temperature of 48°C. Gas production ceased almost immediately on raising the temperature above 48°C and the digestion process appeared to stop.

Samples of sludge were withdrawn periodically during this trial and laboratory tests were conducted on the withdrawn sludge to determine its pH level, and alkalinity. The pH level and alkalinity found remained constant throughout the entire run indicating that both stages of the anaerobic digestion process were affected. A build up of volatile acids would result had the liquefaction stage

continued and the gasification stage been affected. The build up of volatile acids would certainly have caused a decrease in bicarbonate alkalinity. Since neither occurred it was quite evident the whole process had ceased. The digester contents were discarded when no noticeable reaction appeared evident after 12 days.

In the third trial, the temperature was raised to 48°C and maintained at this temperature for a week. It was anticipated that sufficient thermophiles would have developed after this time so that the temperature could be raised slowly without hindering digestion.

The digester operating at 48°C was producing sludge gas although the volume produced was quite low. The sludge gas was checked for carbon dioxide content and found to contain approximately 50 per cent carbon dioxide. The small volume of gas produced and the large carbon dioxide content indicated retarded digestion.

The temperature at which the digester was operating was raised to 49°C, after operating at 48°C for one week, at which time gas production ceased. Again all biological action appeared to stop and after ten days the digester contents were discarded.

Tests performed for pH level and alkalinity in all of these cases indicated values which were within the desirable range (respectively 6.6 to 7.6 and 2000 to 5000 ppm). pH levels varied from 6.9 to 7.1 and values found for

alkalinity were about 3000 ppm in all cases. No apparent biological reactions were evident, even after a week. Tests conducted to check on biological action indicated that the pH level and alkalinity did not change noticeably, thus verifying the fact that no biological reactions were taking place.

Thermophilic digestion was not achieved by the previously mentioned methods as outlined. Past reports were checked for procedures used in developing a high rate thermophilic bacterial culture. The procedure used by Malina [10] is as follows.

Malina [10] filled his experimental digesters with a mixture of 30 per cent digested sludge and 70 per cent raw activated sludge. The temperature of this mixture was raised to 52.5°C. Gas production became evident after approximately 20 days indicating that thermophilic digestion had been achieved.

The same procedure as used by Malina [10] was attempted as the other methods had failed. A mixture made up of approximately 70 per cent raw activated sludge and 30 per cent digested sludge was put in the experimental digesters. The digesters were placed in water baths whose temperature was maintained at 52.8°C. Gas production became apparent on the tenth day, indicating thermophilic digestion had been achieved. One of the digesters was fed some raw sludge on the fifteenth day. When it became

apparent after the second feeding that no ill effect was experienced by this digester, all digesters were fed and a daily maintenance program was set up. The digesters were all fed at a low organic loading rate for the first four days. After this initial period, the loading rates were increased slowly until the desired rates were achieved. Once the desired loading rate was achieved, the digester was fed at this rate for ten days. Gas production was found to be constant by the end of ten days indicating stabilization had occurred. Tests were conducted on the digested sludge for the next 15 days and the results of these tests recorded. No noticeable changes occurred during this period thus indicating the system had stabilized.

The second phase of this research project was to alter the retention time from 12 days to 8 days. Changing the retention time from 12 days to 8 days was accomplished by increasing the volume of sludge fed and withdrawn from 300 millilitres per day to 450 millilitres per day. Ten days were allowed for acclimatization after changing the retention time before tests were conducted on the digested sludge. The tests were terminated and the digesters shut down after ten days as there were no noticeable changes in test results.

The three unsuccessful attempts point out that it was not possible to achieve thermophilic digestion by taking sludge digested at a mesophilic temperature and increasing

its temperature into the thermophilic range. The unsuccessful attempts to develop a thermophilic bacterial culture from a mesophilic culture indicates that there must be two distinct temperature zones for growth of organisms as shown by Fair and Moore [6] in Figure 2.

In order to evaluate the performance of an anaerobic sludge digester, studies must be conducted on the combined effects of temperature, loading and retention time on each of the parameters: reduction in volatile matter, gas production, gas quality, volatile acids concentration, pH level and alkalinity.

The pH level has to be considered one of the most important factors that influences the anaerobic digestion process. The digester substrate must be maintained within a pH range of 6.6 to 7.6 for anaerobic digestion to occur although the optimum pH range is from 7.0 to 7.2. The pH level was checked frequently due to its importance in anaerobic digestion.

The graphs in Figures 12, 13, and 14 show that pH levels present in both the mesophilic and thermophilic digesters were within the acceptable range for optimum digestion. It was noted that the pH levels were slightly greater for thermophilic digestion than for mesophilic digestion. The graphs (Figures 12, 13 and 14) include the alkalinities with the pH levels of each digester as pH level and alkalinity are interrelated.

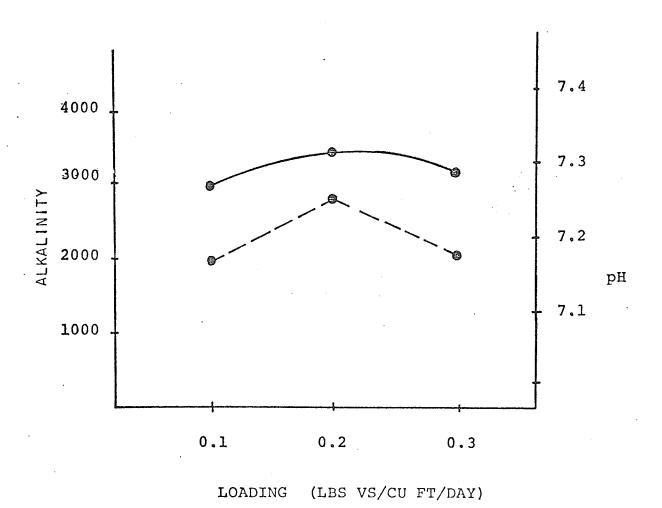


FIGURE 12 ALKALINITY AND PH vs LOADING

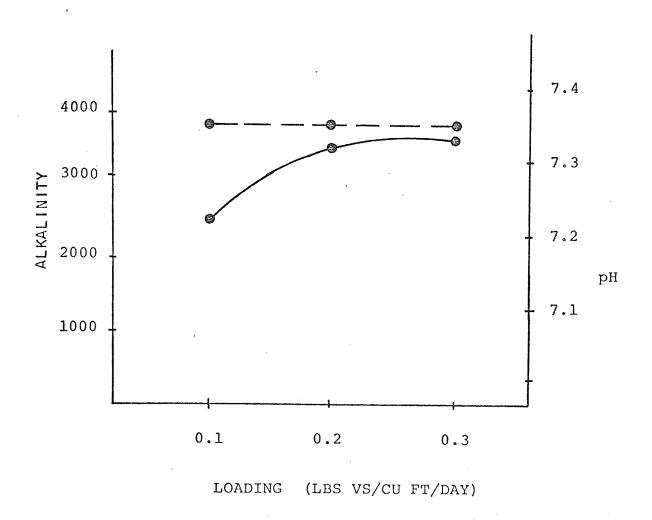


FIGURE 13 ALKALINITY AND PH $_{\mathbf{V}^{\mathrm{S}}}$ LOADING

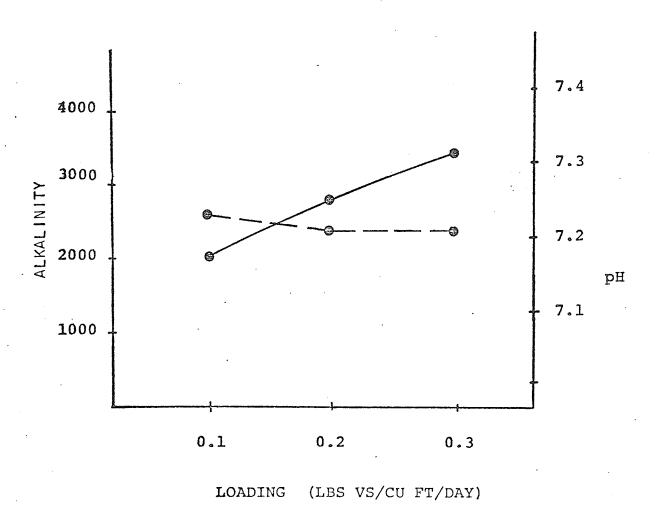


FIGURE 14 ALKALINITY AND PH VS LOADING

The desirable alkalinity range for optimum digestion is between 2,500 and 5,000 ppm. In this alkalinity range, any large increase in volatile acids concentration will be neutralized by the bicarbonate alkalinity resulting in a minimum drop in pH. Total alkalinity is approximately equal to bicarbonate alkalinity under normal anaerobic digestion conditions. The graphs (Figures 12, 13 and 14) show that the alkalinities recorded for all digesters were within the desirable range and thus offered good buffer action for any increase in volatile acid concentration. This good buffer capacity was responsible for maintaining the pH level within the desired range and also eliminated any substantial variation of it.

Being able to maintain a fairly constant pH level in systems employing periodic feeding may be difficult but must be accomplished if optimum digestion is to occur. The acid formers can readjust more quickly to loading variations than can the methane formers, thus volatile acid concentrations increase quite rapidly after feeding in this type of system as indicated by McGee [19]. A large concentration of volatile acids would cause a decrease in pH level if sufficient buffering action due to alkalinity was not available.

The alkalinity remained fairly constant in digesters at different loading rates during mesophilic digestion, as

shown in Figure 12.

During thermophilic digestion, it was noted that alkalinity increased as the loading rate increased.

Alkalinity was found to be higher for the digesters operating at a 12 day retention time than the ones operating at an 8 day retention time.

An explanation for these variations in alkalinity is given by McCarty [1]. He states that at the near neutral pH level of interest for anaerobic digestion, between 6.0 and 8.0, the major chemical system controlling pH is the carbon dioxide - bicarbonate system which is related to pH or hydrogen ion concentration through the following equilibrium equation:

$$[H^{+}] = K_{1} \frac{[H_{2}CO_{3}]}{[HCO_{3}]}$$

The carbonic acid concentration $[H_2CO_3]$ is related to the percentage of carbon dioxide in the digester gas, K_1 is the ionization constant for carbonic acid and the bicarbonate ion concentration $[HCO_3^-]$ forms a part of the total alkalinity in the system. Total alkalinity in the system is composed of both bicarbonate alkalinity and volatile acid alkalinity.

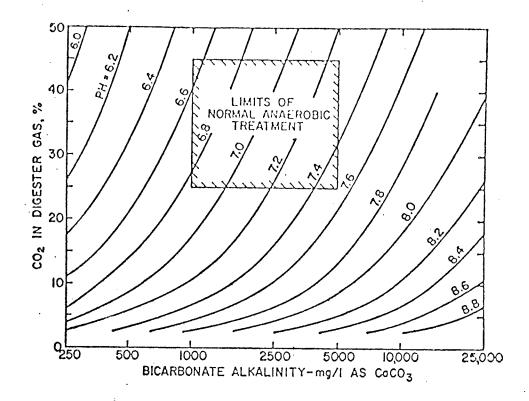


Chart No. 1 RELATIONSHIP BETWEEN pH AND BICARBONATE CONCENTRATIONS NEAR 95° F. (McCarty)

From chart No. 1, the relationship between pH levels and bicarbonate alkalinity becomes apparent. Although this chart was set up for a temperature of 95°F, the curves on this chart would simply be shifted over for different temperatures.

From the chart, it is noted that as the pH level increases, the bicarbonate alkalinity also increases, provided the carbon dioxide content of sludge gas produced remains constant. The bicarbonate alkalinity is part of the total alkalinity therefore as the level of pH increases

so would total alkalinity, provided a constant carbon dioxide content prevailed.

The digester operating at a 12 day retention time was found to have a higher pH level than the digester operated at an 8 day retention time, therefore its alkalinity should be higher as shown by Chart No. 1. Figures 13 and 14 show that the alkalinity is higher for the digester using a 12 day retention time.

The raw sludge collected for feeding the system was kept refrigerated to preserve it for the entire test period. The raw sludge was analyzed prior to feeding and found to contain 65.4 per cent volatile matter. A comparison of the percentage of volatile solids in the raw and digested sludge is shown in Figures 15, 16, 17 and 18. The relationship between volatile solids and time for mesophilic digestion is shown in Figure 15. The graph shows that the digested sludge contained approximately 50 per cent volatile matter for both loading rates.

The graphs (Figures 16, 17 and 18) represent a comparison of the per cent volatile solids present in the raw and digested sludge for thermophilic digestion. The graphs also show that the per cent of volatile solids present in the digested sludge increased as the loading rate was increased at both retention times. The per cent volatile solids present in the digested sludge for the three different loading rates varied approximately two per cent

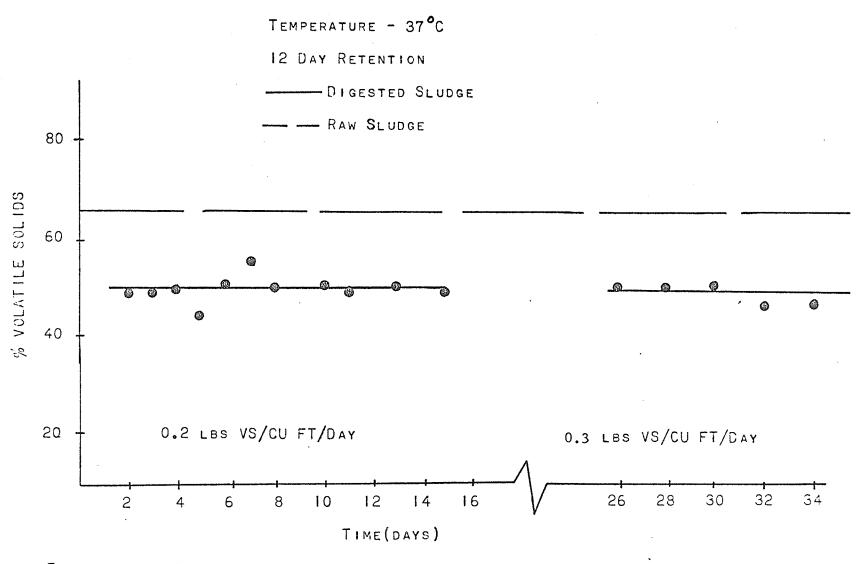


FIGURE 15 % VOLATILE SOLIDS VS TIME

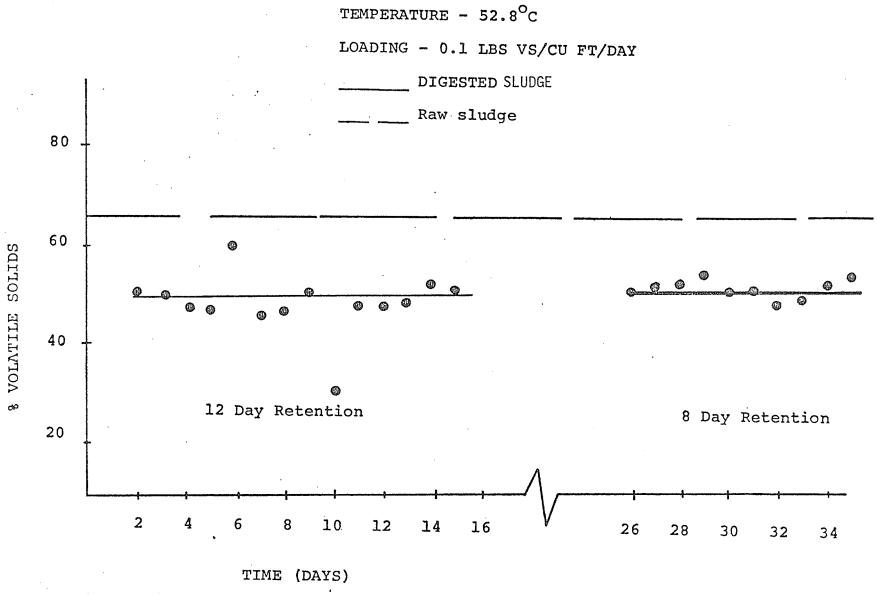


Figure 16 % volatile solids vs time

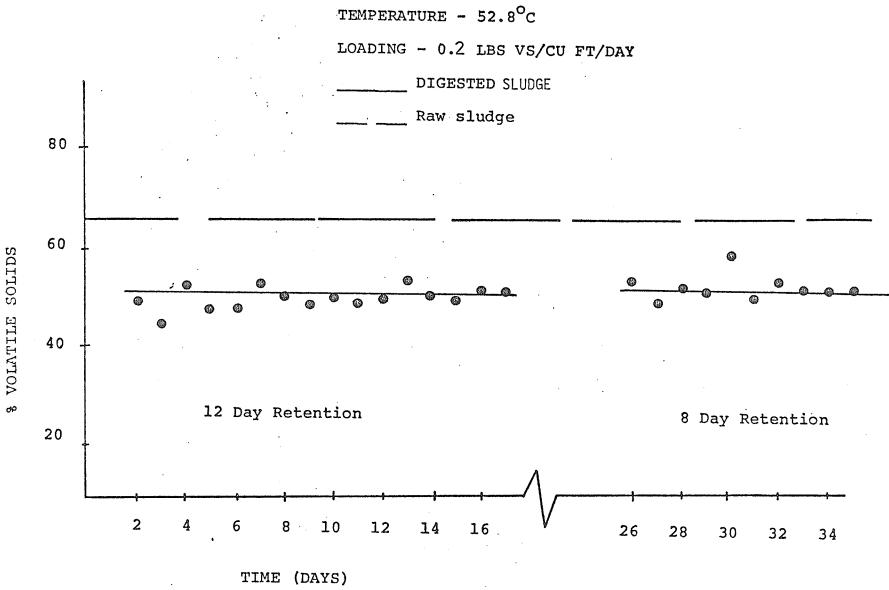


Figure 17 % volatile solids vs time

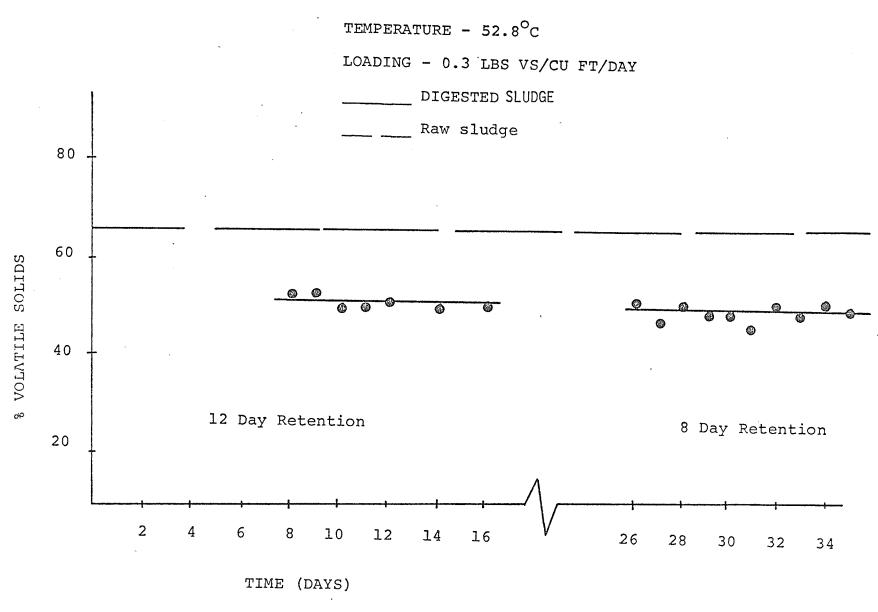


Figure 18 % volatile solids vs time

TEMPERATURE - 37°C

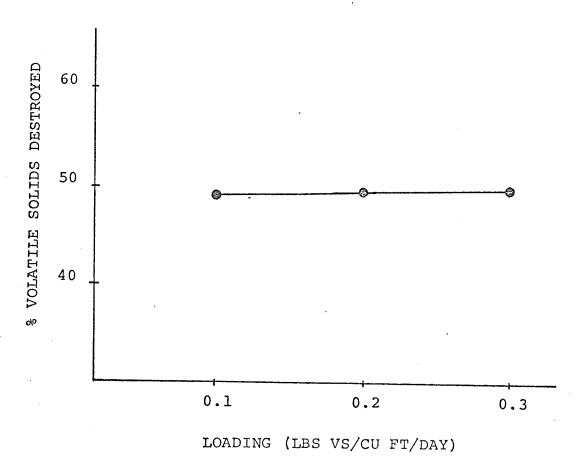


FIGURE 19 % VOLATILE SOLIDS DESTROYED vs LOADING

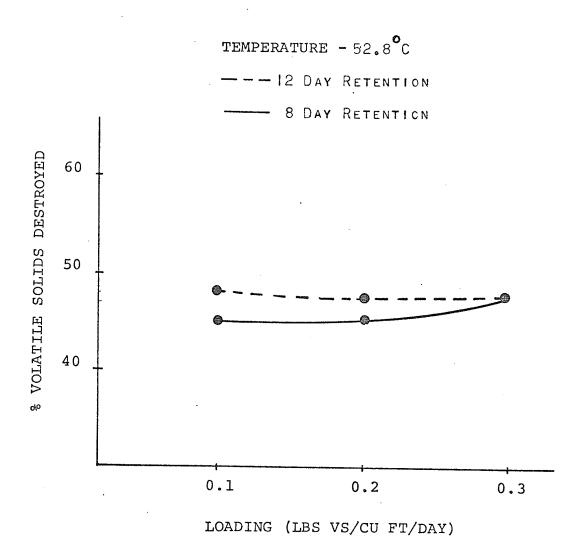


FIGURE 20 % VOLATILE SOLIDS DESTROYED vs LOADING

for the digesters operated at a 12 day retention time. The difference between percentage of volatile solids present in the digested sludge for corresponding loading rates at 12 and 8 day retention times for thermophilic digestion was negligible.

The relationship between the percentage of volatile solids destroyed and loading rates are shown in Figures 19 and 20. This relationship for mesophilic digestion is shown in Figure 19. The percentage of volatile solids destroyed as indicated in Figure 19 was approximately 48 per cent for all loading rates, although it was slightly higher as the loading rate was increased.

The thermophilic case is shown in Figure 20 which indicates that the percentages of volatile solids destroyed at a retention time of 8 and 12 days were similar. The percentage of volatile solids destroyed at the 12 day retention time varied from 48, 47 and 47 per cent, respectively, for loading rates of 0.1, 0.2 and 0.3 pounds of volatile solids per cubic foot of digester per day. For the 8 day retention time, the percentage of volatile solids destroyed varied frome 46, 46 and 47 per cent, respectively, at loading rates of 0.1, 0.2 and 0.3 pounds of volatile solids per cubic foot per day.

Figures 21, 22 and 23 represent the effect of loading on the reduction of volatile solids. The grams of volatile matter destroyed per day increases as the loading rate

increases for both thermophilic and mesophilic digesters. The amount of volatile matter removed was slightly larger for the thermophilic digesters operated at a 12 day retention time than for the ones operated at an 8 day retention time. The amount of volatile matter removed per day varied slightly for corresponding loading rates in both mesophilic and thermophilic digestion.

The graphs (Figures 21, 22 and 23) show that the amount of volatile matter destroyed was proportionately greater at loading rates of 0.1 and 0.3 pounds of volatile solids per cubic foot per day than at a loading of 0.2 pounds of volatile solids per cubic foot per day for both the mesophilic and thermophilic digesters. This was also evident from the curves given in Figures 19 and 20, as the percentage of volatile matter destroyed was less at a loading rate of 0.2 pounds of volatile solids per cubic foot per day than at either of the other two rates. This phenomenon was more pronounced in thermophilic digestion than in mesophilic digestion.

The relationship between the loading rate and the volume of sludge gas produced for mesophilic digestion is given in Figure 24 while Figures 25 and 26 represent this relationship for thermophilic digestion. The volume of gas increased as the loading rate increased in all cases. The volume of gas produced was almost identical for the mesophilic digester and the two thermophilic digesters at

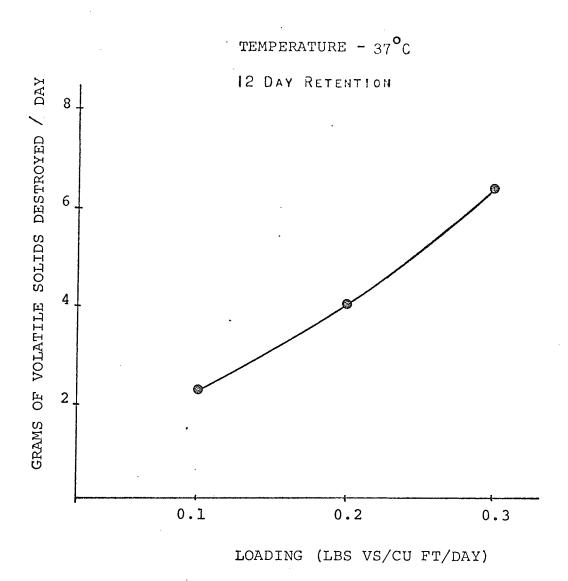


FIGURE 21 GRAMS OF VOLATILE SOLIDS DESTROYED PER DAY vs LOADING

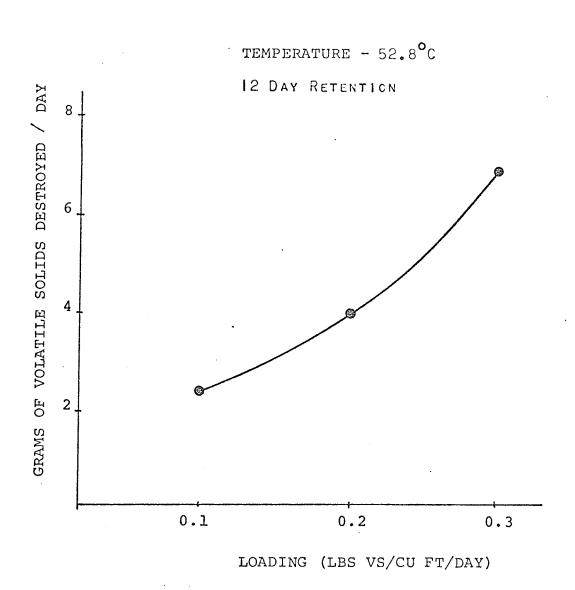


FIGURE 22 GRAMS OF VOLATILE SOLIDS DESTROYED PER DAY VS LOADING

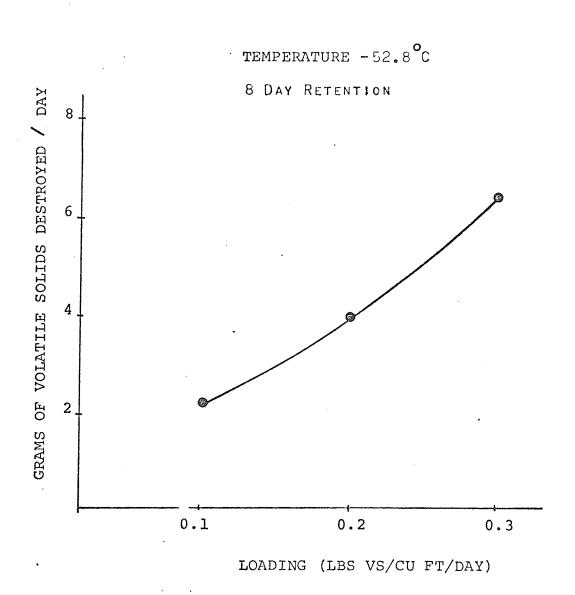


FIGURE 23 GRAMS OF VOLATILE SOLIDS DESTROYED PER
DAY vs LOADING

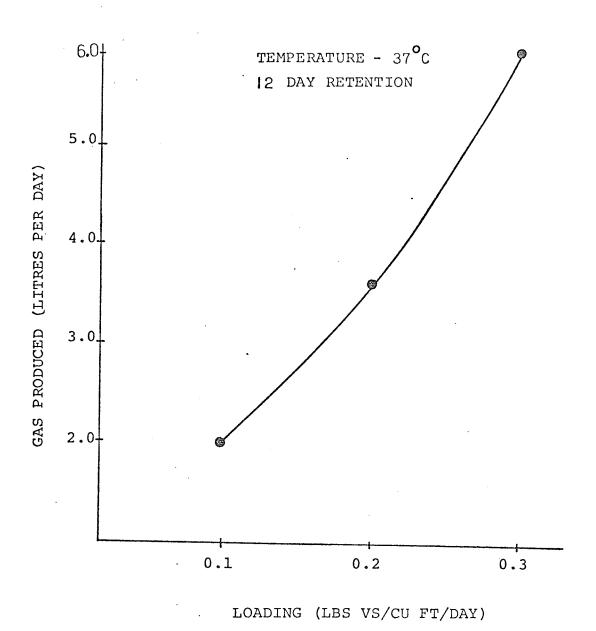


FIGURE 24 GAS PRODUCED VS LOADING

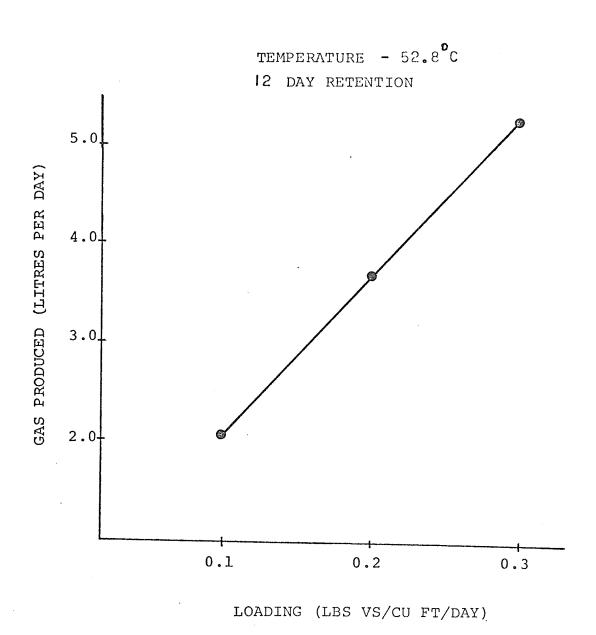
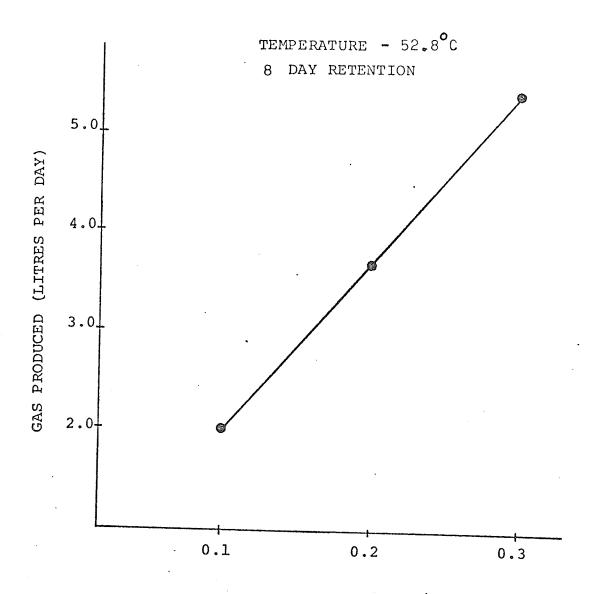


FIGURE 25 GAS PRODUCED VS LOADING



LOADING (LBS VS/CU FT/DAY)

FIGURE 26 GAS PRODUCED VS LOADING

loading rates of 0.1 and 0.2 pounds of volatile solids per cubic foot per day. The volume of gas produced at a loading rate of 0.3 pounds of volatile solids per cubic foot per day, was almost the same at an average of 5.3 litres per day for both thermophilic digesters. The mesophilic digester had an average gas production of 6.1 litres per day at this loading which is substantially higher than either of the thermophilic digesters.

Figures 27 and 28 represent the relationship between volatile solids destroyed per day and gas produced per day. This relationship for mesophilic digestion represented by Figure 27 indicates that at the higher loading rates, more gas was produced for a given amount of volatile solids destroyed. This is further substantiated by Figure 29, which represents the relationship between volumes of gas produced per gram of volatile solids destroyed and loading rate. It is apparent from this graph that the volume of gas produced per gram of volatile solids destroyed increased as the loading rate was increased.

Figure 28 represents the relationship between the volume of gas produced and amount of volatile solids destroyed at thermophilic digestion. The graph (Figure 28) indicates that as the retention time is increased, reduction in volatile matter increases slightly, but the volume of sludge gas produced does not increase proportionately.

The relationship between gas produced per gram of

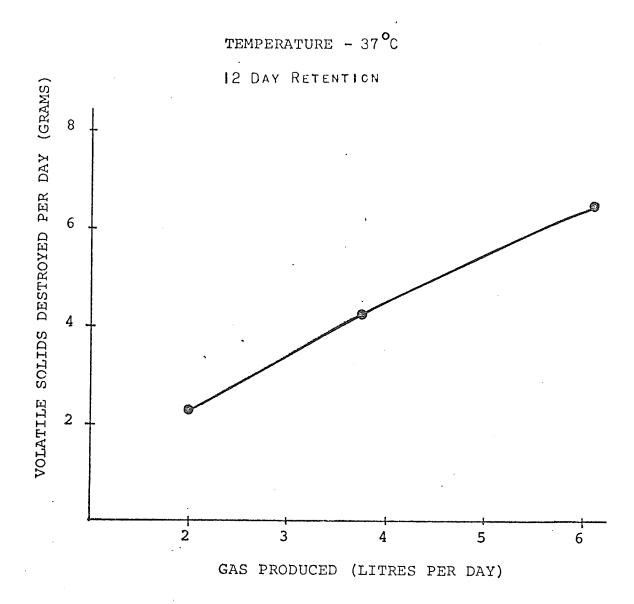


FIGURE 27 VOLATILE SOLIDS DESTROYED PER DAY vs GAS PRODUCED

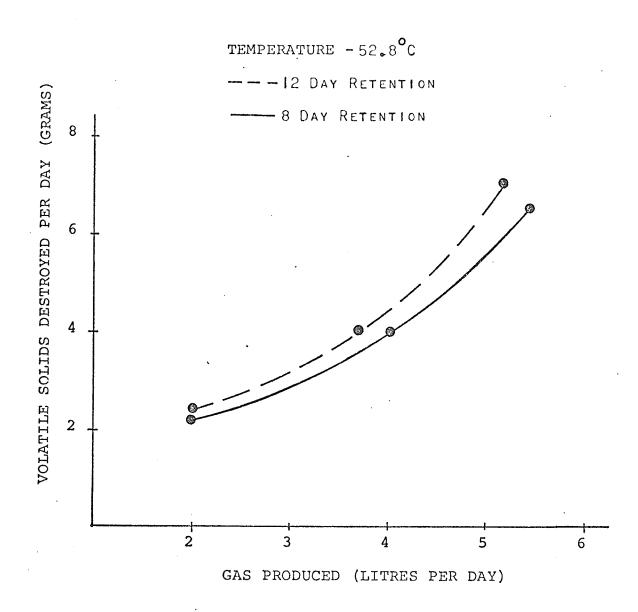
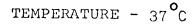


FIGURE 28 VOLATILE SOLIDS DESTROYED PER DAY VS GAS PRODUCED



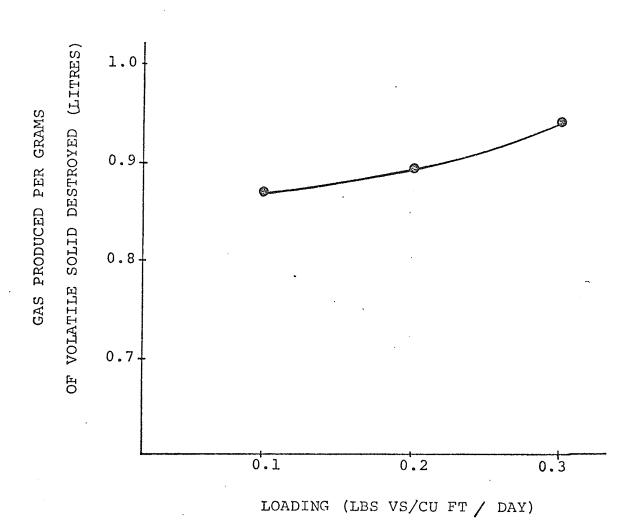


FIGURE 29 GAS PRODUCED PER GRAM OF VOLATILE SOLID

DESTROYED VS LOADING

volatile solids destroyed and loading rate for thermophilic digestion was shown in Figure 30. The graph indicates that the volume of gas produced per gram of volatile solids destroyed was slightly greater for an 8 day retention time than one of 12 days.

Figures 31, 32 and 33 represent the relationship between loading rate and per cent carbon dioxide present in the sludge gas. The carbon dioxide content of the sludge gas was within the desired limits (30 to 35 per cent carbon dioxide) for normal digestion for all digesters.

Sludge gas from the mesophilic digesters was analyzed and found to contain 30 per cent carbon dioxide at the two lower loading rates and 35 per cent carbon dioxide at a loading rate of 0.3 pounds of volatile solids per cubic foot per day.

The thermophilic digesters operating at a 12 day retention time produced results similar to those of mesophilic digestion. The carbon dioxide content found for 12 day retention time was 29, 31 and 34 per cent, respectively, for loading rates of 0.1, 0.2 and 0.3 pounds of volatile solids per cubic foot per day. The carbon dioxide content of the sludge gas in the eight day retention case was 30, 34 and 34 per cent, respectively, for loading rates of 0.1, 0.2 and 0.3 pounds of volatile solids per cubic foot per day. The higher carbon dioxide

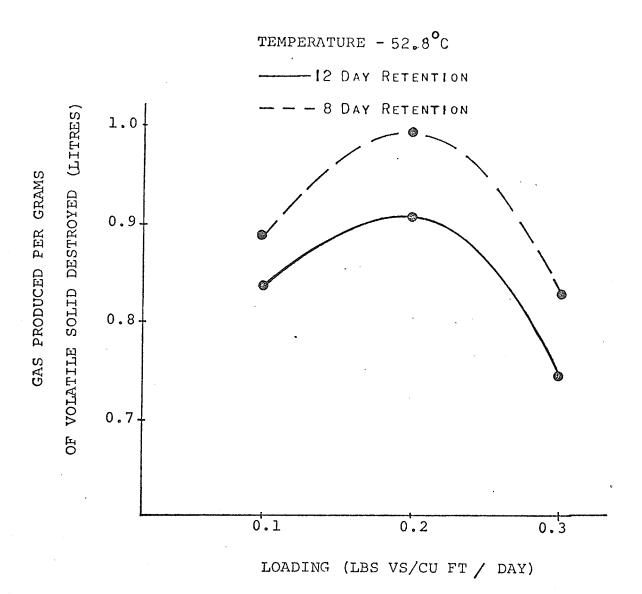


FIGURE 30 GAS PRODUCED PER GRAM OF VOLATILE SOLID DESTROYED vs LOADING



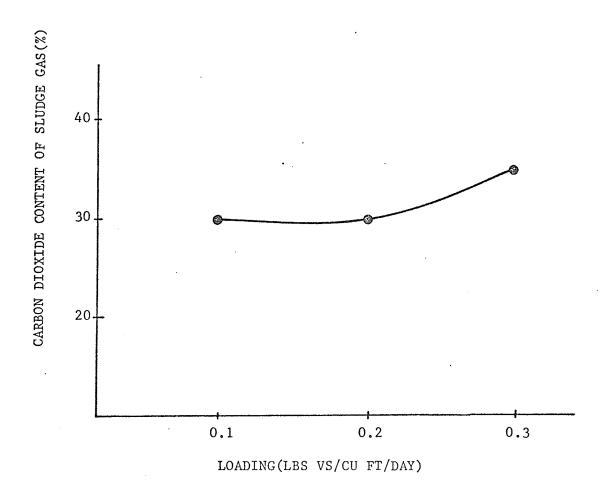
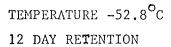


Figure 31 CARBON DIOXIDE CONTENT OF SLUDGE GAS vs LOADING



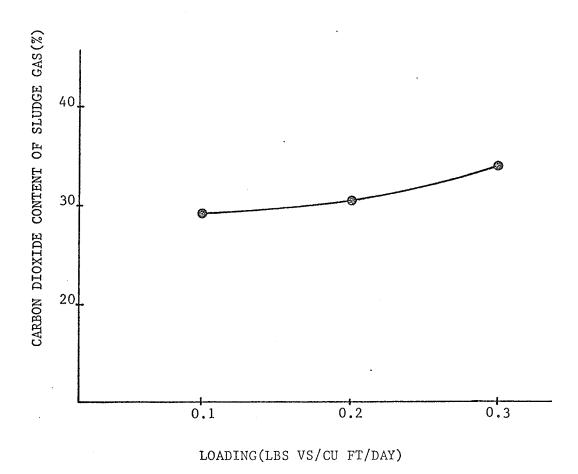


Figure 32 CARBON DIOXIDE CONTENT OF SLUDGE GAS vs LOADING

TEMPERATURE - 52.8°C
8 DAY RETENTION

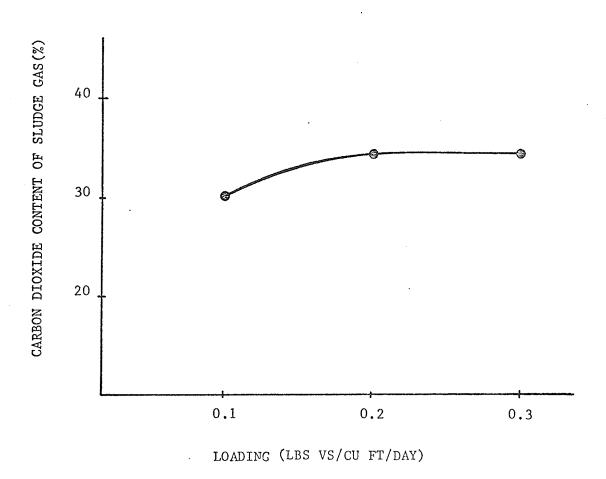


Figure 33 CARBON DIOXIDE CONTENT OF SLUDGE GAS vs LOADING

contents experienced at the high loading rates indicated that the anaerobic digesters process was not functioning as efficiently at these loadings.

The relationship between volatile acids and loading rates for thermophilic digestion operating at an 8 day retention time was represented by the graph (Figure 34). The relationship in Figure 34 indicates that the volatile acid concentration increased as the loading rate increased but its concentration was well within desirable limits (less than 5,000 ppm as indicated by McCarty [1]) for all cases.

Figure 35 gives an indication of the volatile acid concentration present in both mesophilic and thermophilic digestion at a loading rate of 0.3 pounds of volatile solids per cubic foot per day.

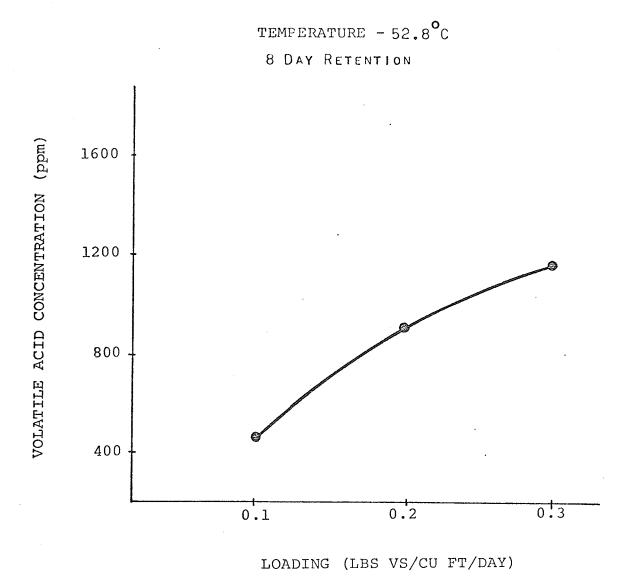
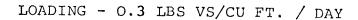
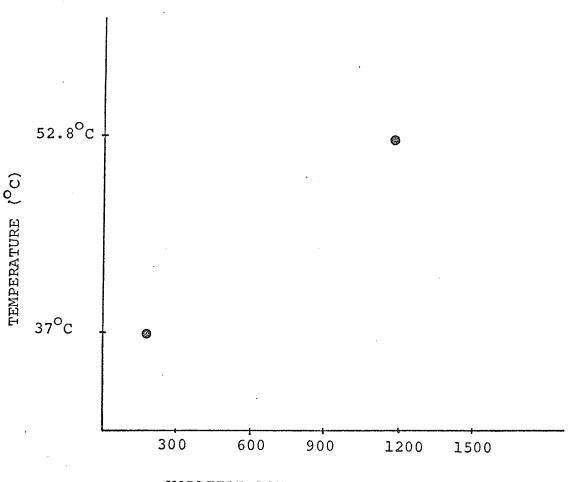


FIGURE 34 VOLATILE ACID CONCENTRATION vs LOADING





VOLATILE ACID CONCENTRATION (ppm)

FIGURE 35 VOLATILE ACID CONCENTRATION VS TEMPERATURE

CHAPTER 8

CONCLUSIONS

The performance of mesophilic and thermophilic anaerobic sludge digesters was evaluated by studying the combined effects of temperature, loading and retention time on each of the following parameters: reduction in volatile matter, gas production, gas quality, volatile acids, pH level and alkalinity.

The data gathered during this investigation indicated that similar results were obtained for mesophilic and thermophilic digesters operated at comparable retention times and loading rates.

The percentage reduction in volatile matter during thermophilic digestion was slightly higher for a digester operated at a 12 day retention time than one operated at an 8 day retention for comparable loadings. The amount of volatile matter removed varied directly as the loading rate for mesophilic and thermophilic digesters. The results were similar for mesophilic and thermophilic digesters at comparable loadings although at a 12 day retention time the amount of volatile solids removed in thermophilic digesters was slightly higher than at an 8 day retention

time.

Gas production was found to be equal for mesophilic and thermophilic digestion at comparable loadings of 0.1 and 0.2 pounds of volatile solids per cubic foot of digester per day. Gas production at a loading of 0.3 pounds of volatile solids per cubic foot of digester per day was the same for thermophilic digestion at the 12 and 8 day retention times but the mesophilic digester had a substantially larger gas production at this loading. Gas production increased directly as the loading increased for mesophilic and thermophilic digesters.

The carbon dioxide content of sludge gas was approximately 30 per cent for mesophilic and thermophilic digesters operating at loading rates of 0.1 and 0.2 pounds of volatile solids per cubic foot of digester per day, but at a loading rate of 0.3 pounds of volatile solids per cubic foot per day the carbon dioxide content of sludge gas was approximately 35 per cent for all digesters.

The pH level was higher for thermophilic digestion than for mesophilic digestion. The pH level remained the same for the different loading rates used during thermophilic digestion, but the pH level was greater for digesters operated at a 12 day retention time than for those using an 8 day retention time.

The alkalinity range for mesophilic digestion increased slightly as the loading rate was increased. The

alkalinity range for thermophilic digestion increased directly as the loading rate was increased but this increase was more pronounced at the 8 day retention time than for the 12 day retention time.

The volatile acids concentration was greater for thermophilic digestion than for mesophilic digestion. The volatile acids concentration increased directly as the loading rate was increased.

The digested sludge from both mesophilic and thermophilic digesters gave off a disagreeable odor but the intensity of odor detected was the same for all digesters.

The thermophilic organisms were found to be much more sensitive to temperature variations than the mesophilic organisms. A drop of 2°C for less than two hours resulted in gas production being approximately halved for thermophilic digestion. The temperature varied between 36°C and 38°C during mesophilic digestion without any noticeable effects on digestion.

The phenomenon of thermophilic digestion was achieved by raising a mixture of 30 per cent digested sludge and 70 per cent raw activated sludge to a temperature of 52.8°C. Gas production commenced in 10 days indicating that thermophilic digestion had been achieved.

The results obtained in this study indicated that no benefits were derived from thermophilic digesters operated at comparable loading rates and retention times

as mesophilic digesters, when digester temperature was 37°C for mesophilic digestion and 52.8°C for thermophilic digestion.

BIBLIOGRAPHY

- 1. McCarty, P.L. (1964). "Anaerobic waste treatment fundamentals," Public Works, Vol. 95. Part 1. Chemistry and Microbiology, pp. 107-112. Part 2. Environmental Requirements and Control, pp. 123-126. Part 3. Toxic Materials and Their Control, pp. 91-94. Part 4. Process Design, pp. 95-99.
- Kormanik, R.A. (1968). "A resume of the anaerobic digestion process," Water and Sewage Works, Vol. 115, pp. R154-R166.
- 3. Baker, H.A. (1956). "Biological formation of methane," Industrial and Engineering Chemistry, Vol. 48, pp. 1438-1443.
- 4. Pohland, F.G. and D.E. Bloodgood (1963). "Laboratory studies on mesophilic and thermophilic anaerobic sludge digestion," W.P.C.F., Vol. 35, pp. 11-42.
- 5. W.P.C.F. Manual of Practice No. 16. "Anaerobic sludge digestion."
- 6. Fair, G.M. and E.W. Moore (1934). "Time and rate of sludge digestion and their variation with temperature," Sewage Works Journal, Vol. 6, pp. 3-13.
- 7. Garber, W.F. (1954). "Plant scale studies of thermophilic digestion at Los Angeles," Sewage and Industrial Wastes, Vol. 26, pp. 1202-16.
- 8. Buswell, A.M. (1957). "Fundamentals of anaerobic treatment of organic wastes," Sewage and Industrial Wastes, Vol. 29, pp. 717-721.
- 9. Heukelekian, H. (1933). "Digestion of solids between the thermophilic and non-thermophilic range," Sewage Works Journal, Vol. 5, pp. 757-762.

- 10. Malina, Jr., J.F. (1961). "The effect of temperature on high rate digestion of activated sludge,"

 Proceedings of the Sixteenth Industrial Waste Conference, Purdue University, May 1961, pp. 232-250.
- 11. Rowe, D.R. (1971). "Anaerobic sludge digestion, mesophilic-thermophilic," Water and Sewage Works, Vol. 118, pp. 74-76.
- 12. Golueke, G.F. (1958). "Temperature effects on anaerobic digestion of raw sewage sludge," Sewage and Industrial Waste, Vol. 30, pp. 1225-1232.
- 13. Malý, J. and H. Fadrus (1971). "Influence of temperature on anaerobic digestion," W.P.C.F., Vol. 43, pp. 641-650.
- 14. Hill, D.J. and E.D. Schroeder (1969). "The methane fermentation between mesophilic and thermophilic temperature ranges," Water and Waste Engineering, Vol. 6, pp. 46-49.
- 15. Torpey, W.H. (1955). "Loading to failure of a pilot high rate digester," Sewage and Industrial Wastes, Vol. 27, pp. 121-133.
- 16. Rudolfs, W. and H. Heukelikian (1930). "Thermophilic digestion of sewage solids," Industrial and Engineering Chemistry, Vol. 22, No. 1, pp. 96-99.
- 17. Shindala, A. and T.G. Byrne (1970). "Anaerobic digestion of thickened sludge," Public Works, Vol. 101, pp. 73-76.
- 18. Standard Methods for the Examination of Water and Wastewater. Thirteenth Edition.
- 19. McGhee, T.J. (1971). "Volatile acid concentration in batch fed anaerobic digesters," Water and Sewage Works, Vol. 118, pp. 130-.32.
- 20. Bloodgood, D.E., Personal Correspondence.
- 21. Sawyer, C.N. and P.L. McCarty (1967). Chemistry for Sanitary Engineers. Second Edition, McGraw-Hill Inc., New York.
- 22. Hach D R Colorimeter Methods Manual. Seventh Edition.

APPENDIX

DATA SUMMARY

Mesophilic Digesters

Temperature - 37°C

Solids Retention Time - 12 days

Loading			
(lbs. VS/cu. ft./d.)	0.1	0.2	0.3
Gas (1/d)	2.05	3.73	6.1
рН	7.13	7.24	7.16
Alkalinity (ppm)	3000	3400	3200
TS (gm/l)	36.62	32.38	40.42
VS (gm/1)	18.88	16.10	19.78
% VS	50.2	49.9	49.2
% VS Reduction	47	48	48
% CO ₂	30	30	35

Thermophilic Digesters

Temperature - 52.8°C

Solids Retention Time - 12 days

	Loading		
lbs.	VS/cu.		

(lbs. VS/cu. ft./d.)	0.1	0.2	0.3
Gas (1/d)	2.08	3.68	5.2
рН	7.35	7. 35	7.35
Alkalinity (ppm)	2540	3417	3525
TS (gm/l)	19.82	28.76	31.44
VS (gm/1)	9.14	14.80	15.90
% VS	48.7	50.9	50.4
% VS Reduction	48	47	47
% CO ₂	29	31	34

Thermophilic Digesters

Temperature - 52.8°C

Solids Retention Time - 8 days

Loading	•		
(lbs. VS/cu. ft./d.)	0.1	0.2	0.3
Gas (1/d)	2.0	3.90	5.4
рН	7.22	7.21	7.21
Alkalinity (ppm)	2025	2800	3475
TS (gm/1)	14.00	24.52	34.75
VS (gm/1)	7.16	12.67	17.33
% VS	51.2	51.1	49.4
% VS Reduction	46	46	47
% CO ₂	30	34	34

ADDITIONAL READING

- Thiel, P.G. (1969). "The effect of methane analogues on methanogenis in anaerobic digestion," Water Research, Vol. 3, No. 3, pp. 215-223.
- Siebert, M.L. and D.F. Toerien. (1967). "The proteolytic bacteria present in the anaerobic digestion of raw sewage sludge", Water Research, Vol. 3, No. 4, pp. 241-250.
- Anaerobic Digestion: D.F. Toerien and W.H.J. Hatlingh
- (a) (June 1969). "The microbiology of anaerobic digestion", Water Research, Vol. 3, No. 6, pp. 385-416.
- (b) (July 1969). "The characterization and control of anaerobic digestion", Water Research, Vol. 3, No. 7, pp. 459-494.
- (c) (August 1969). "Kinetics of anaerobic fermentation", Water Research, Vol. 3, No. 8, pp. 545-558.
- (d) (September 1969). "The applications of the process in waste purification", Water Research, Vol. 3, No. 9, pp. 623-644.
 - Van Huyssteen, J.J. (1970). "The determination of short-chain fatty acids in aqueous solution by gas-liquid chromatography", Water Research, Vol. 4, No. 10, pp. 645-659.
 - Moore, Stephen F. and Edward D. Schroeder. (1970).

 "An investigation of the effects of residence time on anaerobic bacterial denitrification", Water Research, Vol. 4, No. 10, pp. 685-694.

Population Description of the Non-methanogenci phase of Anaerobic Digestion:

- (a) (February 1970). "Isolation, characterization and Identification of numerically important bacteria", Water Research, Vol. 4, No. 2, pp. 129-148.
- (b) (April 1970). "Hierarchical classification of isolates", Water Research, Vol. 4, No. 4, pp. 285-304.
- (c) (April 1970). "Non-hoerarchical classification of isolates by principal component analysis", Water Research, Vol. 4, No. 4, pp. 305-314.
- (d) (April 1970). "Intermediary metabolism of non-methanogenic bacteria", Water Research, Vol. 4, No. 4, pp. 315-326.

- Pretorius, W.A. (1971). "Anaerobic digestion of raw sewage", Water Research, Vol. 5, No. 9, pp. 681-688.
- Novak, John T. and Dale A. Carlson. (1970). "The kinetics of anaerobic long chain fatty acid degradation", W.P.C.F., Vol. 42, No. 11, pp. 1932-1943.
- Speece, R.E. and Jan A. Kem. (1970). "The effect of short-term temperature variations on methane production", W.P.C.F., Vol. 42, No. 11, p. 1990.
- Dague, Richard R. (1968). "Application of digestion theory to digestion control", Vol. 40, No. 12, pp. 2021-2032.
- Lynam, Bart, Gerald McDonnell and Max Krup. (1967).
 "Start-up and operation of two new high-rate digestion systems", W.P.C.F., Vol. 39, No. 4, pp. 518-535.
- Radharkrishnan, I., S.B. De and B. Nath. (1969). "Evaluation of the loading parameters for anaerobic digestion of cane molasses distillery wastes", W.P.C.F., Vol. 41, No. 11, Part 2, pp. R431-R440.
- Lawrence, Alonzo W. and Perry L. McCarthy. (1969).

 "Kinetics of methane fermentation in anaerobic treatment", W.P.C.F., Vol. 41, No. 2, pp. Rl-Rl7.
- Sanders, F.A. and Don E. Bloodgood. (1965). "The effect of nitrogen to carbon ratio on anaerobic decomposition", W.P.C.F., Vol. 37, No. 12, pp. 1741-1752.
- Kugelman, Irwin J. and Perry L. McCarty. (1965). "Carbon toxicity and stimulation in anaerobic waste treatment", W.P.C.F., Vol. 37, No. 1, pp. 97-116.
- Dague, Richard R., Ross E. McKinney and John T. Pfeffer. (1966). "Anaerobic activated sludge", W.P.C.F., Vol. 38, No. 2, pp. 220-226.
- Spence, R.E. and Jan A. Kem. (1970). "The effect of short-term temperature variations on methane production", W.P.C.F., Vol. 42, No. 11, pp. 1990-1997.
- Novak, John T. and Dale A. Carlson. (1970). "The Kinetics of anaerobic long chain fatty acid degradation", W.P.C.F., Vol. 42, No. 11, pp. 1932-1943.
- Miholets, Ernest M. and Joseph F. Malina Jr. (1968).
 "Effect of amino acids on anaerobic digestion",
 W.P.C.F., Vol. 40, No. 2, Part 2.

- Pfeffer, John T., Martin Leiter and John R. Worlund. (1967). "Population dynamics in anaerobic digestion", W.P.C.F., Vol. 39, No. 8.
- Dague, Richard R., Ross E. McKinney and John T. Pfeffer. (1966). "Anaerobic activated sludge", W.P.C.F., Vol. 38, No. 2.
- McCarty, Perry L. and Ross E. McKinney. (1961). "Volatile acid toxicity in anaerobic digestion", W.P.C.F., Vol. 33, No. 3, p. 223.
- McCarty, Perry L. and R.E. McKinney. (1961). "Salt Toxicity in anaerobic digestion", W.P.C.F., Vol. 33, No. 4, p. 399.
- Albertson, Orris E. (1961). "Ammonia nitrogen and the anaerobic environment", W.P.C.F., Vol. 33, No. 9.
- Cookson, John T. and N.C. Burbank Jr. (1965). "Isolation and identification of anaerobic and facultative bacteria present in the digestive process", W.P.C.F., Vol. 37, No. 6, p. 822.
- Torpey, Wilbur N. and Norman R. Melbinger. (1967).

 "Reduction of digested sludge volume by controlled recirculation", W.P.C.F., Vol. 39, No. 9, pp. 1464-1474.
- Zablatzky, H.R. and Stanley A. Peterson. (1968). "Anaer-obic digestion failures", W.P.C.F., Vol. 40, No. 4, pp. 581-585.
- Dague, Richard R. (1968). "Application of digestion theory to digester control", W.P.C.F., Vol. 40, No. 12, pp. 2021-2032.
- Buzzell, James C. Jr. and Clair N. Sawyer. (1963). "Bio-chemical vs physical factors in digester failure", W.P.C.F., Vol. 35, No. 2, pp. 205-221.
- Keefer, C.E. and Hans C. Urtes. (1962). "Digestion of volatile acids", W.P.C.F., Vol. 34, No. 6, pp. 592-604 and Vol. 35, No. 3, pp. 334-343, March 1963.
- Ludzack, F.J., R.B. Schaffer and M.B. Ettinger. (1961). "Temperature and feed as variables in activated sludge performance", Vol. 33, pp. 141-156.
- American Society of Civil Engineers, Journal of the Sanitary Engineering Division:
 - Loehr, Raymond C. (1966). "Design of anaerobic digestion systems", Vol. 92, No. SA1, pp. 19-29.

- Schroeder, Edward D. (1966). "Mass and energy relationships in anserobic digestion", A.S.C.E., Vol 92, No. SAL, pp. 85-98.
- Quon, Jimmie E. and Glen M. Johnson. (1966). "Drainage characteristics of digested sludge", A.S.C.E., Vol. 92, No. SA2, pp. 83-96.
- Andrews, John F. (1966). "Dynamic model of the anerobic digestion process", A.S.C.E., Vol. 95, No. SA1, pp. 95-116.
- Sawyer, Clair N. and J.S. Brumbling. (1960). "Fundamental considerations in high rate digestion", Vol. 86, No. SA3, pp. 49-64.
- Malina, J.F. Jr. (1964). "Thermal effects on completely mixed anserobic digestion", Water and Sewage Works, Vol. 111, pp. 52-56.
- Langford, Leonard L. (1958). "Digester net effective capacity", Vol. 105, p. 435.
- Andrews, J.F., J.F. Thomas and E.A. Pearson. (1964).
 "Determination of volatile acids by gas chromatography", Water and Sewage Works, Vol. 111, pp. 206-210.
- Hinden, E., R.S. May, R. McDonald and G.H. Dunston. (1964). "Analysis of volatile fatty acids in sewage by gas chromatography", Water and Sewage Works, Vol. 111, pp. 92-95.
- Eckenfelder, W. Wesley Jr. (1967). "Mechanisms of sludge digestion", Water and Sewage Works, Vol. 114, pp. 207-210.
- Pohland, F.G. (1964). "High rate digestion", Water and Sewage Works, Vol. 111, pp. 262-265.
- Garrison, Walter E., John D. Parkhurst and Carl A. Nagel. (1964). "Accelerated sludge digestion, Part 2 Gas recirculation natural, artificial", Water Works and Waste Engineering, Vol. 1, No. 2, pp. 58-63.
- Symons, G.E. (Chairman) (1950). "Sludge digestion tank heating and related problems", A panel discussion, Sewage and Industrial Wastes, Vol. 22, No. 1.
- Mueller, H.F., A.M. Buswell and T.E. Larson. (1956).
 "Chromatographic determination of volatile acid",
 Sewage and Industrial Wastes, Vol. 28, No. 3, p. 255.

- Grune, Werner N. (1956). "Development of a continuous ass chromatographic analyzer for sludge digestion studies", Sewage and Industrial Wastes, Vol. 28, No. 12, p. 1433.
- Mau, Gordon E. (1956). "Applying recent research to design of separate sludge digesters", Sewage and Industrial Wastes, Vol. 28, No. 10, p. 1199.
- Alexander, Aleck. (1956). "Chemical control of digester scum", Sewage and Industrial Wastes, Vol. 28, No. 7, pp. 931-934.
- Faust, S.D., H.E. Orford and W.A. Parsons. (1956).
 "Control of sludge volumes following lime neutralization of acid wastes", Vol. 28, No. 7, p. 872,
- Lohmeyer, George T. (1959). "A review of sludge digestion", Sewage and Industrial Wastes, Vol. 31, pp. 221-235.
- Mueller, Lee E., Ervin Hinden, Jesse V. Lunsford and Gilbert H. Dunstan. (1959). "Some characteristics of anaerobic sludge digestion, (1), Effects of loading", Sewage and Industrial Wastes, Vol. 31, No.6, pp. 669-677.
- Chmielowski, Jerry, James R. Simpson and Peter C.G. Isaac. (1959). "Use of gas chromatography in sludge digestion", Sewage and Industrial Wastes, Vol. 31, pp.1237-1258.
- Fischer, A.J. and R.A. Greene. (1945). "Plant scale tests on thermophilic disestion", Sewage and Industrial Wastes, Vol. XVII, No. 4.
- Buck, T.C., Jr. C.E. Keefer and Hester Halch. (1953). Bacteriological Studies of Sludge Digestion
 - a) "A facultative anserobic isolated from digested sludge", Sewage and Industrial Wastes, Vol. 24, No. 9, pp.993-1002.
 - b) "Optimum pH at which sludge digests when inoculated with streptococcus diploidus", Sewage and Industrial Westes, Vol. 25, No. 10, pp. 1174-1178.
 - c) "Effect of different quantities of streptococcus diploidus on sludge direction", Seware and Industrial Wastes, Vol. 25, No. 12, pp. 1406-1413.
- Mosey, F.E. (1971). "The toxicity of cadmium to anaerobic digestion: its modification by inorganic anions", W.P.C.F., Vol. 70, No. 5.

- Smith, Paul H. (1965). "Pure culture studies of meth-anogenic bacteria", Purdue University, pp. 583-588.
- Woods, Calvin. (1965). "Determination of proteins in waste water", Purdue University, pp. 501-515.
- Gardy, F.J.A., R.D. Cole and E.A. Pearson. (1963).
 "Enzyme activity as a parameter of digester performance", Purdue University, pp. 51-65.
- McCarty, Perry L. and Marc. H. Brosseau. (1965). "Effect of high concentrations of individual volatile acids on anaerobic treatment", Purdue University, pp. 283-296.

Books and Articles:

- "Progress in industrial microbiology", Vol. 9, edited by D.J.D. Hockenhull, MA, Ph.D., F.I. Biol. J. and A. Churchill, London WlH 4AE 104 Gloucester Place, CRC Press.
 - "Anaerobic digestion in biological waste treatment", E.F. Kirsch and R.M. Sykes, p. 155.
 - "Lab and design procedures for waste water treatment", W.W. Eckenfelder Jr. and Davis L. Ford. The Centre of Research in Water Resources, University of Texas, EHE-10-6802, CRWR-31.
- International Association of Water Pollution REsearch 1969.
- Advances in Water Pollution Research Proceedings of the Fourth International Conference held in Prague, 1969, edited by S.H. Jenkins.
- The succession of microbial processes in the anaerobic decomposition of organic compounds", Jiri Hausler, p. 407.
- Advances in Water Pollution Research Proceedings of the International Conference held in London, September 1962, edited by W.M. Eckenfelder.

Articles:

- (a) "Recent work on the treatment and dewatering of sewage sludge", J.D. Swanwick, F.W. Lussignea and R.C. Baskerville, p. 387.
- (b) "Determination of the size of tanks for the aerobic and anaerobic degradation of organic wastes", Franz Popel, p. 451.

"Advances in biological waste treatment", edited by W.W. Eckenfelder Jr. and Brother Joseph McCabe.

Article:

"Gas chromatographic studies of sewage metabolism", R.R. Kountz, J.B. Nesbitt and Charles Forney, Jr. p. 85.

Recent Progess in Microbiology, edited by N.E. Gibbons, 1962.

Article:

"General aspects of amino acids fermentation in Japan", J. Asai, p. 325.