HIGH-SOLIDS ANAEROBIC FERMENTATION OF POULTRY MANURE

A Thesis

Presented in Partial Fulfillment of the Requirements for the Degree Master of Science

bу

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Approved By

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LIST OF ABBREVIATIONS

CH₄ Methane gas

CO₂ Carbon dioxide

 H_2S Hydrogen sulfide

NH₃ Ammonia gas

NH₄ Ammonium ion

CH₃COOH Acetic acid

TS Total Solids (%)

TVS Total Volatile Solids (%)

TKN Total Kjeldahl Nitrogen (% d.b.)

 NH_3-N Ammonia represented as Nitrogen (% d.b.)

TOC Total Organic Carbon (% d.b.)

VSD Volatile Solids Destroyed (% d.b.)

COD Chemical Oxygen Demand (mg/1)

d.b. dry basis

1 liters

lb/ft³ pound per cubic foot

ppm parts per million

C/N Carbon to Nitrogen ratio

S/F Seed to Feed ratio

P.M. Poultry manure

C.S. Corn stover

M.S.W. Municipal sewage waste

H.S.F.R. High-solids fermenter residue

USDA United States Department of Agricultural

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Chapter I

INTRODUCTION

Anaerobic fermentation is a natural and commonly used treatment for stabilization of organic waste. In the treatment, decomposition of organic matter occurs in the absence of molecular oxygen. If the process of decomposition is carried to completion, the end products are stabilized organic compounds and bio-gas (CH $_4$ and CO $_2$). The organic compounds can be used as a soil fertilizer while the bio-gas is a non-polluting source of energy. For several decades the process of anaerobic digestion has been applied to agricultural waste management. The conventional method for anaerobic digestion requires a dilute waste, generally 90% to 95% moisture content, while most bio-mass, crop residue has 50% to 90% TS and animal manure has 15% to 35% TS. Addition of dilution water incurs three major problems:

- * larger volume to be handled in the treatment
- * considerably high energy input to maintain the mesophilic or thermophilic temperature condition for biological activities inside the fermenter

* additional water increases the potential for non-point pollution.

All these requirements would make the conventional process economically less attractive for poultry manure. The process may result in a net negative energy yield when used to treat the waste from a large scale facility. A process which can treat the high-solids waste in "as produced" state is preferred to make the treatment more energy efficient and economical. High-solids fermentation also referred to as "dry" fermentation is a process in which the decomposition occures in the absence of free water.

The feasibility of achieving efficient methane production from organics at high solids content, mainly crop residue, has been reviewed by Jewell et al. (11). Jewell defined the anaerobic "dry" fermentation process as the fermentation of organics at solid concentrations higher than that at which water will drain from the substrate. A small laboratory scale fermenter showed efficiency of conversion of a mixture of wheat straw and dairy cow manure with initial solids at 25% very close to that in a 10% solids mixture (11). The results of the subsequent work on a large scale reactor fermenting crop residues at 25% - 30% initial solids content was also very encouraging. A $110 \, \mathrm{m}^3$ reactor using wheat straw and dairy manure at 25% initial solids content was designed and operated successfully for 100 days at Cornell State University.

Poultry manure is generally collected in a dry condition. Depending on the manure collection technique employed, the total solids content of the manure varies from 10% to 70%. The higher values corresponds to the use of litter material for keeping the manure in dry condition. Thus conventional anaerobic digestion of poultry manure requires a large quantity of water to dilute it to 10% or less total solids content before digestion. Simple calculations can show that dilution of poultry manure collected at 55% total solids to 10% total solids would need water which would give over three times the original mass. This additional water would not only increase the size of reactor to accommodate the added water but also would require a large quantity of heat to keep the slurry temperature high enough for bacterial activities. If the same manure is used at 20%total solids for anaerobic digestion, the water requirement could be reduced by 60% . If it is treated at 30% total solids, the water required for dilution would be only one third the quantity of the manure. Wujcik (27) in his study on batch reactors with dairy cow manure observed very little difference in methane production at 10% initial solids and 30% initial solids.

The use of poultry manure at high solids content for anaerobic fermentation seems logical but has a potential problem. The high nitrogen content of the manure is likely

to cause ammonia toxicity and ultimately the failure of the treatment facility.

The objective of this research was to evaluate the use of poultry manure, mixed with agricultural crop residues as a source of carbonaceous matter, at high initial total solids for anaerobic fermentation. Corn stover being readily available and having a relatively high C/N value (50-90) was mixed with poultry manure. The effect of three different solid contents and the inoculum on gas production was studied. No documentation was found on the high-solids anaerobic digestion of poultry manure.

Chapter II

OBJECTIVES

The purpose of this research was to evaluate the possible use of a high-solids anaerobic fermentation process as an alternative to conventional slurry-type anaerobic digestion for bio-gas production from poultry manure. The specific objectives of the study were as follows:

- 1. Determine the effects of initial total solids content on bio-degradation of the substrate.
- 2. Evaluate the effects of type and quantity of seed material used to initiate the process of bio-gas production.
- 3. Indicate the optimum detention time of the process.
- 4. Compare the bio-gas production rates and total energy out put with the conventional process.

Chapter III

LITERATURE REVIEW

Poultry production in U.S. has expanded greatly during the last three decades. The total number of chickens produced annually has increased from 630 million in 1950 to 3.6 billion in 1979 (18). There are about 200,000 egg producing farms in the country. The USDA estimates that 85% of the market eggs are produced by only 12,000 large producers, just 6% of the total. The expansion has also brought in a trend in the poultry industry to raise a large number of birds at one location. Broiler production in U.S. is concentrated in relatively few southern states. The ten leading states in broiler production produce about 83% of the total production. Four large companies share about 18% of the total production.

The largest single problem associated with an intensive, confined production is manure handling and disposal. The techniques generally employed for poultry waste management are:

- * storage then spreading on crop lands
- * aerobic and anaerobic lagoons
- * composting

* anaerobic digestion

The normal way of utilizing the manure is to store it in the poultry house in pits beneath the cages and then spread it on crop land. However for a large size operation at one location spreading of manure requires larger areas than are usually available. Studies have shown that about four tons of fresh manure may be spread on a acre of land devoted to corn production with out causing any nitrogen toxicity in the soil. Thus 100,000 hens would require about 1,600 acres of land per year (19).

Lagoons have been tried with some success but they frequently have had serious odor problems.

Composting of poultry manure mixed with bedding material has resulted in odorless, fly-free environments. Although this process offers an opportunity to recover and reuse a portion of the nutrient in the waste, the overall benefits are too small to make the process attractive for agricultural waste management. In general it has been observed that under normal agricultural conditions, the cost of applying compost to the land has been greater than the benefits received (13).

Anaerobic digestion presents a unique method of treating the poultry waste. It not only reduces the pollution level of the waste material but also supplies energy in the form of bio-gas, about 60% to 70% of which is methane.

3.1 ANAEROBIC DIGESTION

The process of stabilizing organic compounds biologically in the absence of oxygen is anaerobic digestion. Most naturally occurring organic matter can be digested anaerobically. The advantages offered by this process are as follows (6,13):

- * higher loading rates than are possible for aerobic treatment
- * 50% to 70% reduction in organic content, thus the digested material represents a lesser pollutional hazard
- * useful end products such as well stabilized organic residue and combustible gases which have commercial value
- * alteration of water-binding characteristics to permit rapid dewatering of the residue
- * solids reduction permits easier material handling after treatment
- * process yields little energy for microbial growth hence less organic material is converted to new cells. It is possible to treat waste that have levels of nutrients which are marginal for aerobic treatment.
- * stabilized digested waste has a lower odor level.

 Anaerobes, responsible for anaerobic digestion, obtain their energy for decomposition of organic matter by utilizing compounds other than dissolved oxygen.

3.1.1 Kinetics of Anaerobic Digestion

Anaerobic treatment of organic wastes is a controlled biodegradation of the substrate in which energy and nutrients are made available to the microorganisms with a portion of the organic material converted to methane and carbon dioxide (6). The entire process of fermentation is described as a three step process involving

- 1. Hydrolysis of complex materials
- 2. Acidogenesis of simple soluble organics
- 3. Methanogenesis of organic acids as shown in Figure 1.

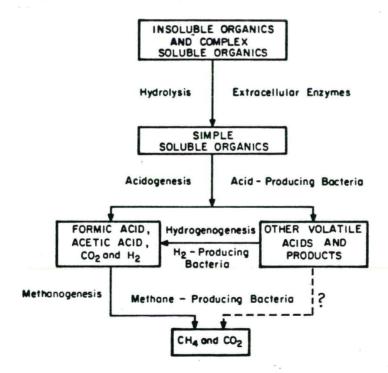


Figure 1: Multi-step nature of anaerobic operations. (Grady & Lim, (6))

During the first step, particulate organics are solubilized and soluble organic compounds are reduced in size to facilitate their transport across cell membrane. The reactions responsible for these process are usually hydrolytic and are catalyzed by the enzymes produced by the bacteria.

In the second step, the acid forming bacteria, consisting of facultative and anaerobic bacteria, oxidize the soluble organic compounds to simple volatile acids such as acetic, propionic, and butyric acids. The process is known as acidogenesis. Some of the acid forming bacteria are capable of utilizing volatile acids larger than acetic acid as well as reduced organic compounds produced by other bacteria, to produce acetic acid, carbon dioxide and hydrogen gas. These bacteria are known as hydrogen producing bacteria and the process is referred to hydrogenogenesis. The combined group of acid and hydrogen producing bacteria is generally called non-methanogenic bacteria and they primarily produce acetic acid, carbon dioxide and hydrogen.

In the third stage, methanogenic bacteria utilize the products of the non-methanogenic phase to produce methane and carbon dioxide. Although it is possible that methane producing bacteria exist which are able to convert other volatile acids and organic end products to methane, none have been isolated (6). Hence that path to methane production is shown by a dotted line in Figure 1. The methanogen-

ic bacteria are obligate anaerobes and can produce methane in two major ways (16):

* by cleavage of acetic acid to methane and carbon dioxide, as shown in Equation 1

$$CH_3COOH \longrightarrow CH_4 + CO_2$$
 (1)

* by reducing carbon dioxide with hydrogen gas to form methane and water, as shown in Equation 2

$$4H_2 + CO_2 \longrightarrow CH_4 + 2H_2O \tag{2}$$

The methane gas has low solubility in water and is rapidly discharged from the system resulting in stabilization of the waste. At standard temperature and pressure, production of 5.62 ft^3 of methane results from the stabilization of one pound of COD in waste material (6). Anaerobic digestion of animal wastes produces gas containing 60% to 70% methane when constantly high rates of digestion are maintained. Loehr (13) reported approximately four to nine cubic foot of bio-gas production per pound of volatile solid added to the digester when poultry, beef and hog wastes were digested. The total estimated manure production and bio-gas production from anaerobic treatment for various animals is shown in table Table 1. The higher amount of gas production per 1b of VS added as in the case of poultry manure indicates the presence of more biodegradable organics in that material than the others.

Table 1

Estimated Manure and Bio-Gas Production

(from Animal Waste per 1000 lb Live Weight)

	airy Cattle	Beef Cattle	Swine	Poultry
Manure Production (1b/day)	85	58	50	59
Total Solids (1b/day)	10.6	7.4	7.2	17.4
Volatile Solids (1b/day)	8.7	5.9	5.9	12.9
Digestive Efficier (% of VS)	ncy 35	50	5 5	6.5
Bio $\frac{1}{3}$ gas Production ft $\frac{3}{1000}$ VS added ft $\frac{3}{1000}$ lb per d	4.7	6.7 39.5	7 • 3 4 3 • 1	

Source: Morris et al. (17)

3.1.2 Process Controlling Parameters

Temperature, pH, organic loading rate, available nutrients, influent solids concentration and toxicity are considered as the major parameters that control the biological process of anaerobic digestion. The effect of these parameters on the rate of digestion have been studied by many investigators. Table 2 summarizes the favorable conditions for optimum performance of the anaerobic reactor.

It has been shown in kinetic studies that the non-methanogenic bacteria have much higher maximum specific growth rate than do the methanogenic bacteria. Consequently non-methanogens can respond more rapidly to environmental stress developed in the reactor than the methanogens. The activities of methanogens is impaired by any sudden changes in the digester environment which may cause its failure. It is importent to maintain stable reactor conditions and any changes, if required, should be made at a rate which can be tolerated by methanogenic bacteria.

 $\begin{array}{c} \textbf{Table 2} \\ \hline \textbf{Range of Controlling Parameters for Effective} \\ \hline \textbf{Digestion.} \end{array}$

Parameter	Optimum level
Temperature	20°C; Psychrophilic process 35°C; Mesophilic process 50°C; Thermophilic process
рН	6.6 to 7.6, although 7.0 to 7.2 is the most suitable range
Organic loading rate	0.2 to 0.4 lb VS/ft ³ -day for poultry manure
Influent solids	10% to 12% solids concentration in case of poultry manure
Toxic conditions	Free ammonia (NH ₃) less than 150 mg/l , depends on pH; Sulfides less than 200 mg/l of soluble sulfides; Oxygen and any highly oxidized material like nitrites and nitrates are not desired at any level
Nutrients	Carbon: Nitrogen should be 20 to 30: 1 and adequate supply of trace elements like Na, Ca, Mg and Fe.

3.1.3 Toxicity

The effects of concentration of a material on the specific growth rate of bacteria are shown in Figure 2. One effect is that as the concentration of a particular material increases, the specific growth also increases and reaches a maximum value at a certain concentration. The range called stimulatory region. On further increase in concentration, no effect is observed on specific growth rate until it reaches the threshold at which it starts decline. At that point, toxicity is occuring and any concentration in excess of that is considered toxic to the microorganisms. The severity of the toxicity increases as the concentration increases from threshold value. Toxicity due to volatile acids, ammonia and sulfide has been frequently observed in cases of improperly functioning anaerobic reactors.

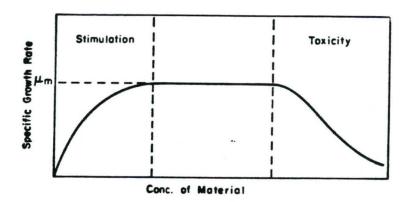


Figure 2: General Nature of Stimulation and Toxicity. (Grady & Lim, (6))

3.1.3.1 Volatile Acids

The effect of volatile acids upon the microorganisms depends on the pH of the medium. When acid forming bacteria produce volatile acids at rates faster than what the methane forming bacteria can utilize then the concentration of volatile acids increases in the reactor. This causes the pH to drop below neutrality. Studies show that when pH is held constant near neutrality, concentrations of up to 10,000 mg/l of acetic or butyric acids have no significant toxic effect upon hydrogen utilizing methanogens (6,15). Whereas propionic acid at the concentration of 1,000 mg/1 exhibits partial toxicity to methanogenic bacteria at neutral pH. Propionic acid also appears to retard the activities of acid forming bacteria in sewage sludge digestion. The inhibition due to propionic acid gets stronger as pH decreases (6), but there is no evidence for such effect with acetic and butyric acids. In general anaerobic reactors operating at neutral pH show little volatile acids toxicity.

3.1.3.2 Ammonia

In the anaerobic system, nitrogen is released as ammonia due to biodegradation of protein. Ammonia in the system is present in either free ammonia, NH_3 , or ammonium ion, NH_4^{\dagger} , depending upon the pH. At higher pH, most of the ammonia is in the free ammonia form. Severe toxicity has

been observed for free ammonia concentrations exceeding 150 mg/l (6). As long as the pH is 7.2 or below, ammonia is in the form of the ammonium ion, which is much less toxic than free ammonia. Concentrations as high as 3,000 mg/l of ammonia as NH_4^{\bullet} can be tolerated with little effect (6). Ammonia in either form is considered to be more toxic to the methanogenic bacteria than to the non-methanogenic bacteria.

3.1.3.3 Sulfide

Sulfates are the major precursors of sulfides in anaerobic treatment units. Sulfides can also result from anaerobic digestion of sulfur containing organic compounds present in the raw wastes (13). At a concentration of 200 mg/l, all inorganic sulfur compounds other than sulfate are found to inhibit methanogenesis in the order as (23):

thiosulfate > sulfite > sulfide > hydrogen sulfide

This toxicity occurs because the sulfate reducing bacteria compete with methane producing bacteria for hydrogen. Although the ability of hydrogen utilizing sulfate reducing bacteria to inhibit the methanogens has been observed, both sulfate reduction and methane production occurs simultaneously in the presence of excess hydrogen (2). Lawrence, et al. (12) reported the concentration of soluble sulfides exceeding 200 mg/l as the cause of methanogenesis inhib-

gested to precipitate the soluble sulfide. These precipitates are highly insoluble and thus reduce the sulfide concentration. Sulfides can also be removed as gaseous hydrogen sulfide in the bio-gas. Therefore to determine the sulfides concentration in the reactor, both the composition of substrate and head space gas should be considered.

3.2 CONVENTIONAL ANAEROBIC DIGESTION OF POULTRY

The process of anaerobic digestion of poultry manure dilute form has been evaluated for methane production and waste stabilization. Laboratory studies on small scale digesters, of the order of several liters, and field studies on large scale digesters, of the order of several cubic meters, have shown potential of poultry manure for methane production. Hill (9) reported biodegradability (Bo, the ratio of volatile solids destroyed to the volatile solids added as detention time tends to infinity) of poultry manure as 0.87 which suggests easier biodegradation of poultry wastes than the wastes of beef and dairy cattle. Hart (7) reported that the anaerobic digestion of poultry manure in one gallon digester operated at mesophilic range of temperature with no mixing yielded 10.7 ft 3 bio-gas per 1b. of volatile matter destroyed. This is about 52% of initial volatile matter destroyed. The methane content of the bio-gas varied from 60% to 70%.

Figure 3, describes the major paths in the process of anaerobic digestion of poultry manure.

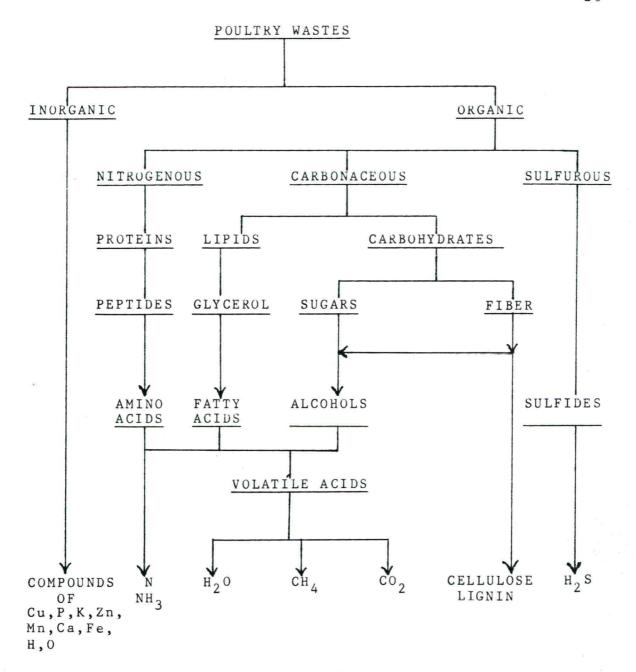


Figure 3: Pathway for Breakdown of Poultry Manure. (Taiganides, (26))

3.2.1 Manure Collection

Manure collection in poultry houses (layers) occurs pit below the cages where manure falls directly from the cages or is scraped from the dropping boards. Pit depth can vary from 0.5 to 8.0 ft (20) depending on the time the manure is stored in the house and the form, liquid or solid, in which it is handled. In shallow pit operations, about one ft depth, the manure is either flushed from the pit daily or scraped out every one to three days. This type of manure handling system is common in small scale units, raising several hundred birds. The medium depth pits are about two ft deep and manure is scraped at periods from every two weeks to every few months. Proper ventilation and dropping boards are provided to keep the manure in fairly dry condition. With this system manure may also be collected in water to form an "indoor lagoon" that is drained out several times a year. In deep pit operations manure is handled in solid form only. Forced ventilation is provided to keep the manure in dry condition. This system is commonly known as a "high-rise type" and manure is removed from the pits once a year. Large scale facilities, more than 100,000 birds, usually employ high-rise type systems for manure collection.

3.2.2 Characteristics of Poultry Manure

The characteristics of poultry manure vary widely depending on the method used for manure collection. Table 3 summarizes the important properties of poultry manure collected by several different methods.

Table 3

Poultry Manure Characteristics

Collection Method	TS % w.b.	V S	TN	NH3 ^{-N} % d.B	T P	K
Shallow pits Medium-depth pits Deep pits	2 5 4 2 5 5	7 4 6 4	6.1 3.6 3.0	0.6	2 • 2 1 • 6 3 • 3	2.0 2.0 2.3
Liquid handling Litter/Manure	8.2 72	64	8.0	4.6 0.9	2.3	3.0 1.8

Source: Overcash, et al.(20)

A major value of poultry manure is the nitrogen content as fertilizer. A facility with 100,000 birds would produce about 23,600 lb manure every day or 8.6 million lb a year which amounts to 175,000 lb of nitrogen annually. About 90% of this nitrogen is in the form of organic nitrogen while 10% is in ammonia form. Uric acid represents about 50% of

the organic nitrogen, which rapidly converts to ammonia nitrogen on biodegradation of the manure. Besides nitrogen, poultry manure also contains an ample quantity of phosphate and potash. Fresh manure has N:P:K ratio of 3.4:1.1:1. The bio-gas production from poultry manure is higher than that from dairy or beef cattle manure and swine manure. As indicated in Table 1, bio-gas production from poultry manure amounts to 110.9 ft³/1000 lb L.W. per day. Thus 100,000 birds would produce about 44,360 ft³ of bio-gas every day which has about 65% methane. The energy thus obtained would be about 27.7 million BTU/day. This energy can be used for a variety of purposes for the facility and makes it less dependent on conventional energy resources.

The process of anaerobic digestion not only provides the energy in the form of bio-gas but also retains the fertility values of the waste, which gives this process an advantage in treating poultry waste.

3.2.3 The Process

In the conventional type anaerobic digestion of poultry manure the manure is diluted to slurry form by adding water as much as two to three times the volume of the waste. The slurry is fed intermittently into a completely mixed or partially mixed anaerobic digester where it is retained for a predetermined time. The digested slurry in the reactor is replaced by incoming fresh slurry. Biological activities

take place in the reactor which convert a part of the organic matter to bio-gas. The process has been studied to evaluate the effects of different loading rates, retention time, temperature, pH, inhibitory concentration of toxic materials and other process governing parameters on bio-gas production and waste stabilization.

Ch.Aubart and S.Fauchille (1), investigated the continuous anaerobic digestion of poultry manure diluted with water. Experiments were conducted in six liter digesters. The manure was diluted to 3.8, 6.0 and 8.1% total solids concentration. Retention time between four to thirty days was used for each treatment. The maximum bio-gas production of $2.52 \, \mathrm{ft}^3/\mathrm{ft}^3$ per day was observed at 3.8% total solids and four days retention time. The maximum methane content was 68.9%.

Converse et al. (3), evaluated the performance of a field size (3426 ft 3) anaerobic digester for treating poultry waste at 8.8% to 12.8% total solids concentration with a detention time of 30 to 53 days. The feeding rate ranged from 0.09 to 0.13 lb VS/ft 3 per day. The gas production was 0.58 to 0.77 ft 3 /ft 3 per day with methane concentration of 55 to 63%.

Patel (22), reported on the performance of a 780 ft batch type fermenter. Poultry manure from a 500 bird facility was treated, after mixing it with water in a ratio of

1:2, at one month batch cycle. The bio-gas production was 1.41 to 2.12 ft^3 per 1b of manure at 35°C . The energy obtained from the process was used mainly for cooking purposes.

Taiganides (26), estimated that about 0.6 lb of water per bird would be required to reduce the total solids of the manure from 25% to 10%, an optimum solid concentration for digestion. The report also indicates that if the digester is loaded at the rate of 0.2 lb/ft³ per day, the capacity required would be 23 ft³ per cubic foot of waste volume, which is equivalent to 0.37 ft³ of digester volume per hen. The waste from 20,000 hens would require a 7,400 ft³ capacity digester. It was estimated that such a digester would produce 5,400 ft³ of bio-gas per day.

These studies indicate that the dilution of the manure to 90% moisture content for anaerobic digestion is indispensable. No work has been done to determine the possible use of manure at lower moisture contents. The size of reactor and amount of heat energy needed is increased with dilution of the manure. A large quantity of material to be handled after treatment and the liquid nature of the effluent may cause non-point pollution problem on disposal. Additional cost incurred due to these requirements makes the treatment economically less attractive on a large scale basis.

3.3 HIGH-SOLIDS ANAEROBIC DIGESTION

Until recently the treatment of agricultural and animal wastes at high solids, in the "as produced state" has been limited to aerobic composting only. Most of the bio-mass available is in a "dry" state. Utilization of bio-mass in relatively dry condition for anaerobic digestion has some distinct advantages but also has some limitations.

Most of the municipal refuse is disposed of by landfill. A typical municipal solid waste has about 20% to 25% moisture. Bacterial decomposition of organic material at high solids content in a landfill is described as occurring in four stages (23):

- 1. aerobic
- 2. anaerobic
- 3. anaerobic, methanogenic, unsteady
- 4. anaerobic, methanogenic, steady

Completion time for the first three stages varies from 180 days to 550 days depending on the composition of material.

After initial stabilization, decomposition takes place actively for many years.

Methane production from landfills is a well known phenomena. The application of this process to the anaerobic digestion of agricultural wastes has been reviewed by Jewell et al. (11). The study showed that the production of methane from bio-mass at high solid contents may have

significant potential for anaerobic fermentation process on widely varying scales. If the reaction rates achieved in the high-solids fermentation can be increased to the values near or equal to those achieved by conventional slurry type fermentation, the process should be competitive.

Wujcik (27) conducted comprehensive studies on dairy cow manure. He studied the effects of water content on the role of methane production via mesophilic anaerobic fermentation. This study attempted to define the limits that moisture and chemicals, ammonia and other salts, have on the hydrolysis reactions, the acid forming mechanism and methane production. Figure 4 indicates relatively uninhibited methane production for solids content upto 30% of the total weight. In general, it was concluded that neither the rates nor the efficiency of substrate conversion were significantly affected by the solids content as high as 30%.

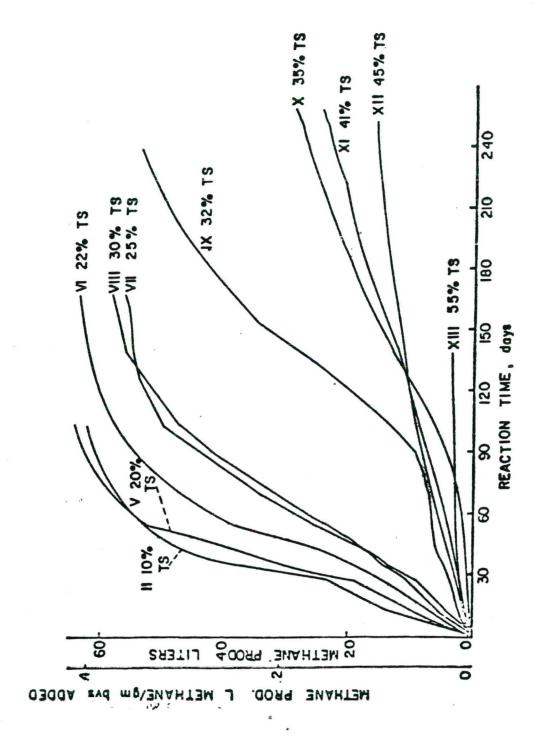


Figure 4: Cumulative Methane Production in a Batch Reactor. (Wujcik, (27)).

3.3.1 Effect of Water Content on Microorganisms

A certain minimum quantity of water in the substrate is required for bacterial activities to be carried out satisfactorily. Unfortunately total water content obtained on drying the sample at 103°C does not account for the impact on microorganisms. This system of measuring water content takes no account of its degree of binding to or association with the dry constituents, which is an important consideration when microbiological factors are to be considered. A concept of water activity is used to determine the effect of water on bacterial metabolism.

The water activity, a_w, of a material is the ratio of vapor pressure of the water in the material to that of pure water at a given temperature (11). It is usually determined by measuring the relative humidity of the atmosphere in equilibrium with the substrate examined. Corry (4) described water activity as a measure of free water in the system. When a solute is mixed with water some of the water becomes associated with the solute molecules. The water activity is reduced and less water is available to microorganisms. The vapor pressure of the solution is reduced. According to Raoult's law, Equation 3, the vapor pressure of a solution relative to the solvent is equal to the mole fraction of the solvent. Thus

$$P = N2$$
 $- = ----- Po = N1 + N2$
(3)

where

P = Vapor Pressure of Solution

Po= Vapor Pressure of Solvent

N1= Moles of Solute

N2 = Moles of Solvent

The ratio P/Po equals to the water activity, $a_{\overline{w}}$, of the system (4).

Most of the microorganisms have difficulty growing below an a_w of 0.6. Bacteria as compared to yeast and mold, are less osmotolerant and their optimum growth rate occurs between water activity values of 0.990 and 0.995. Scott (24), reported the range of a_w for three groups of organisms as:

* bacteria 1.00 to 0.85

* yeast 0.88 to 0.72

* mold 0.76 to 0.62

There are several methods available for measurement of water activity, but generally they involve expensive instrumentation. English (5) established the relationship between water content and water activity for small particles of purified cellulose. Figure 5 shows the value of

 a_w between 0.925 and 0.928 for the substrate solids concentration of 25% to 35%, a typical range at which free water disappears. The water activity of the substrate is in the favorable range for bacterial growth at solids concentration between 25% to 35%. This explains the reason for the small influence of substrate solids concentration up to 30% on methane production as shown in Figure 4.

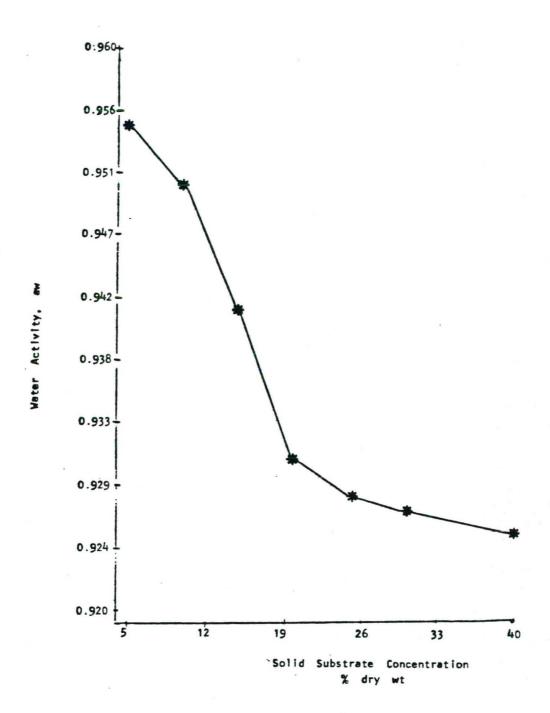


Figure 5: Effects of solids substrate concentration on Water Activity for purefied cellulose. (English, (5)).

3.3.2 Advantages of High-Solids fermentation

The feasibility of achieving efficient methane production from agricultural crop residues at high solids concentration has been reviewed by Jewell at Cornell University (11). A laboratory scale study indicated that the rate and efficiency of conversion of a mixture of straw and dairy cow manure at 25% initial total solids were very close to decay rates in 10% solid mixtures (11). There are several distinct advantages of this process over the conventional wet type fermentation. The advantages are summarized as follow:

- 1. Simple design and operation of the reactor
- The process can produce enough heat during biodegradation of the substrate to maintain the reactor temperature with little or no additional heat inputs in the mesophilic temperature range
- 3. The process can treat many type of organic waste
- 4. Lower labor requirements due to batch set-up
- 5. Little or no additional water requirement
- 6. Minimizes handling and pre-treatment of agricultural products
- 7. Has major pollution control side benefites by producing a final organic residue in a relatively dry state.
- 8. Overall economics are encouraging.

A conventional wet digester for one ton crop residue was hypothetically compared with a dry fermenter for the same quantity of residue (11). It was estimated that the conventional digester would need about 2,100 gal dilution water while the dry fermenter could be operated by adding 790 gal of effluent slurry from a dairy cow manure digester. net energy output from both systems was compared. It was shown that the energy requirements for heating the large quantity of water in the conventional system was as much as 40% to 45% of the gross energy production. The net energy production by dry fermentation was nearly twice the net energy production by the conventional fermentation. Also the steps involving water addition before digestion and water removal after digestion were eliminated in dry fermentation, making the process technically simpler than conventional digestion.

3.3.3 High-Solids Fermentation for Poultry Manur

Poultry manure is normally collected in dry form and it seems logical to treat it by the process of high-solids anaerobic digestion. Due to a high nitrogen content and relatively low carbon to nitrogen ratio (7 to 10), the raw poultry manure anaerobic treatment at a high solids content would be inhibited due to ammonia toxicity. Hashimoto (8) suggested mixing of crop residue with manure for methane fermentation. The major advantage of such mixing is the

nutritional compatibility of highly nitrogenous manure and highly carbonaceous but nitrogen deficient crop residue. Crop residue can be mixed with poultry manure before being digested anaerobically at high solid concentrations. The quantity and type of crop residue required for mixing depends upon the characteristics and availability of the particular residue. Corn stover is available in fairly large quantity and can be a good alternative for mixing with poultry manure. Wheat, rice, or barley straw or any other crop residue can also be used for mixing purposes.

U.S. residue production from corn stover is expected to increase from 101,023 thousand dry tons in 1980 to 107,095 thousand dry tons in 1985 and to 142,536 thousand dry tons by the year 2000 (11). Corn stover collected after harvesting has about 50% total solids content, while 93% to 95% of the total solids are volatile solids and about 65% of the volatile solids are biodegradable. Total nitrogen content is often between 0.5% to 1.0% of the total solids while organic carbon is 45% to 50% of the total solids. Thus the C/N ratio for corn stover is between 50 to 90 which is higher than that for poultry manure. Although some work has been done on dairy cow manure fermentation at high solids content, no background information was found to determine the possible use of poultry manure mixed with crop residue for high-solids anaerobic fermentation.

Chapter IV

EXPERIMENTAL SET-UP

4.1 DIGESTER DESCRIPTION

Plastic buckets with a gasket seal on the lids were used as The diameter of the buckets was 10.5 inches digesters. while the height was 13.75 inches. The total volume of the digester was five gallons (20 liters), about 75% of which was filled with the substrate. Three, 1.5 inch diameter sampling ports were made at 1 inch, 5 inch and 9 inch from the bottom of the bucket. A 1/2 inch nylon nozzle fitting with threads and hose barbs was fixed on the lid of each digester as a gas outlet. Thermocouples were embeded in the substrate at three depths to measure the average temperature inside the reactor. Each reactor was equiped with a separate gas collection line (3/8 inch inside dia. and 1/2 inch outside dia. tygon tubing). Gas from each reactor was collected in a two liter plastic bottle. A reactor with gas collection line and thermocouple attachment is shown in Figure 6.

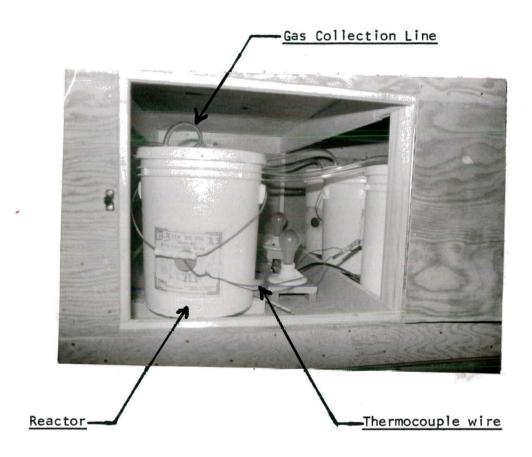


Figure 6: Anaerobic Digester for High-Solids Fermentation

4.1.1 Loading of Reactor

The process was a batch type. The reactors were filled with a mixture of poultry manure, corn stover, seed and water in a predetermined ratio. The reactors were kept in a controlled temperature chamber for the test period without any addition or removal of substrate, except when samples were removed from selected reactors for analyses. Thermocouples were placed at the appropriate depth while loading the substrate.

4.1.2 Temperature Control and Measurement

The substrate in all reactors was kept at 35°C±2°C, the mesophilic temperature range. To maintain this temperature, all the reactors were placed in a 8 ft X 4 ft X 2 ft chamber made from 1/4 inch thick plywood sheet. Four, 100 watt, light bulbs were provided as a heat source. A fan circulated air to maintain a uniform temperature within the chamber. Temperature was monitored by a temperature controler with a YSI temperature probe. A Digistrip-II (KEYE Instrument) recorder was used to read and record the temperature. Figure 7 shows the set-up used to keep the reactors at a constant temperature.

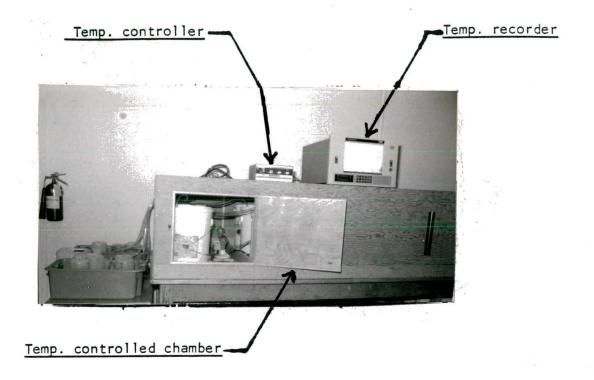


Figure 7: Temperature Controlling and Recording Unit

4.1.3 Gas Collection and Measurement

Each reactor was connected with an individual gas collection unit. Each gas line had a tee with a septum for sampling gas. Jewell (11) reported no significant difference between the use of water and acidified salt solution for the gas collection by liquid displacement method. In this study gas from each reactor was collected in a two liter graduated plastic bottles, initially filled with water, and kept inverted in a tub containing about two inches of water. A syphon system as shown in Figure 8 was arranged

for each tub to keep the water level constant in the tub. This arrangement provide a pressure within the reactor near atmospheric level. The gas flowed through the tygon tubing from the reactor and bubbled up through the water into the storage zone. The collecters were refilled as soon as the entire quantity of water was displaced. The volume of the gas produced was recorded four times a day. Figure 8 shows gas collection from the seven reactors. A similar type of arrangement was provided on the other end of the chamber to collect gas from rest of the reactors.

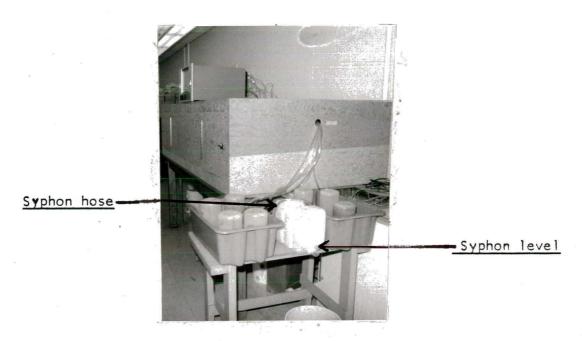


Figure 8: Gas Collection System

4.2 EXPERIMENTAL PROCEDURE

4.2.1 Material Collection and Analysis

The poultry manure for this experiment was collected from the Ohio State University poultry firm, house C. The facility has about 1000 cage layers and manure is collected in a shallow pit underneath the cages. Manure also contained feed material spilled from the feeding trough.

The corn stover was obtained from the South-West branch of the Ohio Agricultural Research and Development Center. It was chopped to 0.5 to 1.0 cm size pieces using a hammer mill before mixing with poultry manure.

Anaerobically digested municipal sludge used for seed was collected from Jackson Pike Waste Water Treatment Plant Columbus, Ohio.

Poultry manure and corn stover were initially analyzed for total solids, volatile solids, total nitrogen and total organic carbon. The procedure used for analysis is described in sec. 4.4.

4.2.2 Sample Collection

4.2.2.1 Poultry Manure

Poultry manure was collected in plastic bags and was stored at room temperature for one week to stabilize the moisture within the material. Samples were taken from each bag at random and composited. These were analyzed to establish initial conditions.

4.2.2.2 Corn Stover

Corn stover was obtained in bales and was shredded to size 0.5 to 1.0 cm. pieces using a hammer mill. The chopped material was stored in the plastic bags at room temperature and samples were taken before it was used for mixing purpose.

4.2.2.3 Municipal Sludge

Anaerobically digested sludge was used to inoculate the starting reactors. The sludge was collected from Jackson Pike WWTP and was refrigerated until it was used. Sample analyses of the sludge were obtained from the plant and the reported total solids, volatile solids and total nitrogen values were used for the required calculations.

4.2.2.4 High-Solids Anaerobic Fermenter Residue

When the high-solids fermenter residue was used as a seed material, samples from the top, middle and bottom layers of the reactor were taken and mixed together. These samples were analyzed without storage before being used as an inoculum.

4.2.2.5 Substrate

After mixing the poultry manure, seed material and corn stover for about 30 minute in a concrete mixer, samples were taken before the substrate was loaded into the reactors. The samples were refrigerated until they were ana-

lyzed. Three weeks later samples were collected from those reactors which had sampling ports. Due to high solids content of the substrate it was difficult to get a representative sample. The results of analyses on this set of samples were not informative. Each reactor was terminated when they stopped producing significant amount of gas. Samples were taken from the top, middle and bottom of the reactors, mixed together and analyzed immediately to establish final parameters.

4.2.2.6 Gas Collection

The gas line from each reactor was provided with a T-joint, one end of which had a septum through which the gas samples were collected using a gas-syringe. The samples were analyzed for methane content immediately.

4.2.3 Criteria to Determine Water Requirement

The poultry manure, corn stover, seeding material and water were mixed together in the proportion to meet the following criterias:

- total solid contents of the substrate; between 30% to
 35% of the wet weight
- 2. the C/N ratio; between 15 and 25
- 3. the poultry manure to corn stover ratio; between two and four

The pre-determined quantity of poultry manure, corn stover and inoculum, either municipal sludge or residue from a high-solids fermenter, were mixed with water in a concrete mixer. The poultry manure while being mixed with corn stover, was observed to form balls, resulting in nonuniform mixing. Better mixing was obtained when poultry manure was first mixed with water and seed, and corn stover added last. The mixing was still not as homogeneous as desired. Due to clumping and ball formation of poultry manure, it was hard to take a representative sample of the substrate.

4.3 TEST SET-UP AND ANALYSIS SCHEDULE

The experiment to evaluate the feasibility of high-solids anaerobic digestion of poultry manure and to study the effect of moisture content on the process was conducted in three stages. In the first stage the major objectives were to evaluate system performance and to obtain acclimated seed for the following experiments. The second and third stage covered different treatments to investigate the effects of substrate moisture content and type and quantity of seed added to initiate the process. The experimental design is explained in the following sections.

4.3.1 Stage I

Two reactors, I-A and I-B, were started with the substrate initial total solids content of 34.0%. The substrate was inoculated with anaerobically digested municipal sludge. The seed to feed (S/F) ratio on a wet basis was 0.5 while the carbon to nitrogen (C/N) ratio was 30.1. The poultry manure was mixed with corn stover in the ratio of 2.5 parts manure to one part stover. The substrate initial volatile solids were 76.6% of the total solids. Gas analysis was not performed in this set of experiments. The only variable observed was gas production. Reactor I-B was terminated after 47 days and the residue was used to inoculate the substrate of stage II. Reactor I-A was operated for 118 days after which it was terminated and the residue was used to inoculate the substrate for the treatment III-D.

4.3.2 Stage II

Three treatments were started at different S/F ratio to study the effects of seed quantity on initialization of the process. Each treatment had two replicates. Residue from the reactor I-B was used to inoculate the substrate. Reactor II-A, II-B and II-C were loaded with substrate at S/F ratio 0.05, 0.13 and 0.26. Initial total solids was 26% to 28%. Due to the dry condition of seed, the mixing of seed and feed material was not complete, which gave inconclusive results. All the reactors were terminated after 71 days and

the residue from reactor II-A2 was used to inoculate the substrate for the treatment III-E.

It was noticed that to use the high-solids fermenter residue as a seed material it is essential to mix the residue with water. The slurry thus obtained should be mixed with the substrate. In order to avoid damage to anaerobic bacteria in the seed, mixing of seed and water should be done without much aeration.

4.3.3 Stage III

A total of five treatments were studied in this stage. Four treatments III-A, III-B, III-C and III-E had three replicates while treatment III-D had only two replicates. Fourteen reactors were initiated at three different solids concentration and two different seeding materials. The initial conditions of the substrate in each treatment are listed in Table 6. With the experience of previous mixing procedures, the substrate was mixed in a slightly different way. The residue from the high-solids fermenter or the municipal sludge was first mixed with a pre-determined quantity of water without much aeration and then this slurry was mixed with poultry manure. Finally corn stover was added. Although a part of poultry manure clumped and formed balls, the distribution of seed material in the substrate was more uniform than in stage II.

The substrate prepared at different initial conditions was loaded into fourteen labeled reactors which were kept inside the constant temperature chamber. The temperature was adjusted so that the substrate in the reactors remained at $35^{\circ}\text{C} \pm 2^{\circ}\text{C}$. All reactors were connected to individual gas collection units. The gas samples were analyzed every week while the substrate was analyzed initially, after 3 weeks and at the end. Because of inability to get representative samples from the sampling ports, the results of intermediate analyses were not significant and are not reported.

After 50 days of operation, all reactors except III-Dl, stopped producing appreciable amounts of gas. These reactors were disconnected from the gas lines and the head space gas in each of them was checked for the presence of ammonia and hydrogen sulfide using a multi-gas detector with DRAGER tubes. The DRAGER tube for ammonia and hydrogen sulfide were inserted into the reactor through the gas outlet and head space gas was drawn through the tubes. The level of ammonia and hydrogen sulfide was recorded.

On termination of the reactors, substrate samples were collected from top, middle and bottom zones. Free water was observed at the bottom of each reactor.

In this set of experiment four reactors suffered from severe leakage and despite initial efforts to seal the

reactors, it was not possible to collect the bio-gas from those reactors. The variables analyzed and the frequency of analyses are listed in Table 4.

Table 4

Analysis Schedule

Parameter	Initial	Daily	Weekly	Final
T.S.	X			X
V . S .	X			X
T . K . N .	X			X
NH_3-N				X
T . O . C .	X			X
рН	X			X
Gas Productio	n	X		
Methane Conte	nt		X	
Temperature		X	k	

4.4 METHODS OF ANALYSIS

High solids concentration of the substrate made it difficult to follow the analysis procedures normally used for liquid waste characterization. The sample preparation for the analysis was the major problem. The test-samples prepared by dilution technique as explained by Jewell (11) were not representative. No other reference was available for the analyses of this type of material. Although Standard Methods for Examination of Waste Water (25) and U.S. Environmental Protection Agency Manual of Methods for Chemical Analysis of Water and Wastes (14) were generally followed to conduct the analysis, some procedures were altered because of the solid nature of the substrate. The procedures used are as follows:

4.4.1 Substrate Analysis

The substrate was analyzed for total solids, total volatile solids, total kjeldahl nitrogen, ammonia nitrogen, total organic carbon and pH.

4.4.1.1 Total Solids

Clean evaporating dishes were pre-heated in muffle furnace at 550° C for about an hour. After cooling in desiccaters, they were weighed and then samples were transferred into marked dishes. The weights of dish plus sample were recorded. The samples were dried at 103° C \pm 1° C

until they reached nearly constant weight, which normally required 22 to 24 hours. After cooling, the final weights were taken and total solids content was determined as

$$TS (%) = ---- * 100$$

$$W2 - W1$$

where

W1 = dish weight (gm.)

W2 = initial weight of sample + dish (gm.)

W3 = weight after oven drying + dish (gm.)

4.4.1.2 Total Volatile Solids

The oven dried samples were ignited at $550^{\circ}\text{C} \pm 50^{\circ}\text{C}$ in a muffle furnace for one and one half hours. The weights before and after ignition were taken and the TVS on a dry basis was calculated as

where

W4 = weight of dish + sample before ignition (gm.)

W5 = weight of dish + ash after ignition (gm.)

4.4.1.3 Total Nitrogen

This test was conducted on a wet sample. Jewell (11) described the soaking-dilution method for sample preparation for the analysis of agricultural crop residue. In that method the substrate was soaked until saturated and after 15 min. of soaking it was centrifuged. The supernatant obtained was used as a sample for analysis. But with the substrate used in this experiment, the samples prepared by that method gave consistently lower results than the results obtained on using raw samples i.e. with out soaking. Five gm of sample was collected from a well mixed substrate and after addition of 400 ml. triple distilled water, one Kel-Pac (Olin-Matheson) and 20 ml. of conc. sulfuric acid, it was digested for one hour. The residue was made alkaline by adding sodium hydroxide-thiosulfate solution and was distilled until 300 ml distillate was collected into 2% boric acid solution. The ammonia in the distillate was determined titrimetrically using mixed indicator. Standard sulfuric acid was used as a titrant. TKN in mg/g of sample was calculated as

$$TKN (mg/g) = A * N * F$$

S

(6)

where

A = ml. of sulfuric acid used for titration

- N = normality of sulfuric acid
 - F = milliequivalent weight of nitrogen, 14 mg.
 - S = sample size, grams.

The total nitrogen was expressed as percent total solids of the substrate.

4.4.1.4 Ammonia Nitrogen

The same technique as TKN determination was used except the digestion step was eliminated. Five gm of sample was mixed with 400 ml. triple distilled water and pH of this solution was adjusted to 9.5 by adding 0.1 N NaOH solution. After adding 25 ml. of borate buffer solution the sample was distilled until 300 ml. of distillate was collected into 2% boric acid solution. Ammonia was then determined titrimetrically. Calculation was similar to that used for TKN determination.

4.4.1.5 Total Organic Carbon

A dry combustion method was used to determine the total organic carbon. Oven dried samples were used for the analysis. The samples were burned at 900°C in the presence of excess oxygen and carbon dioxide produced was absorbed in the ascarite absorption bulb. The weight difference of the bulb before and after analysis gave the amount carbon dioxide liberated and % TOC was determined as

4.4.1.6 pH

pH of the substrate was determined by the procedure normally used for silage (21). In this technique 25 gm of sample
is blended with 225 ml distilled water and pH of the
resulting mixture is measured. For this research, the
substrate and water was mixed using a magnetic stirrer
instead of blending. The pH was determined using an Orian
601-series pH meter equipped with a combination electrode.

4.4.2 Bio-gas Analysis

The volumes of bio-gas produced and collected in the inverted graduated gas collecters were recorded. Readings were taken four times a day for the first two weeks when gas production rate was high and two times a day for the rest of the period. The gas volume was standardized to STP conditions by the following relationship (Ideal gas law):

$$Vstp = (Pa * Va * Tstp)/(Ta * Pstp)$$
 (8)

where

Vstp = bio-gas volume at standard condition, L

Pa = atmospheric pressure, mm Hg

Tstp = standard temperature = 273° K

Ta = ambient room temperature, ${}^{\circ}K$

Pstp = standard pressure = 760 mm Hg

Va = bio-gas volume recorded, L

Bio-gas was analyzed weekly to determine methane content. Figure 9 shows the Gow-Mac series 150 Gas Chromatograph with a 8 X 1/4" Porapak-Q 80/100 mesh column. A thermal conductivity detector was used for gas analysis. Helium gas was used as a carrier gas. Pure methane gas was used to calibrate the GC. The samples were analyzed at constant temperature of 125°C. The area under the methane peak was measured using a planimeter and methane content (%) was calculated as

where

Al = area under methane peak obtained for l ml methane

A2 = area under methane peak obtained for bio-gas sample

S = sample size (m1)

The rest of the bio-gas was assumed to contain mainly carbon dioxide although a small amount of hydrogen sulfide, ammonia and water vapor would be present.



Figure 9: Gow-Mac-150 Gas Chromatograph

Chapter V

EXPERIMENTAL RESULTS AND DISCUSSION

5.1 WASTE CHARACTERISTICS

Poultry manure, corn stover and residue from a high-solids fermenter when used as seed, were analyzed for total solids, volatile solids, total nitrogen and total organic carbon before determining the proportions in which they should be mixed to get the substrate of required initial conditions. Table 5 shows the results of those analyses.

The quantity of poultry manure, corn stover and seed material to be mixed, was calculated using a computer program. The program computed the total solids and C/N values of the mixture containing different proportions of poultry manure, corn stover and seed, and selected the appropriate ratio which would give the desired values of total solids and C/N ratio for the given conditions. The amount of water required was also computed. The S/F ratio in the Stage II was between 0.05 and 0.26 while that in the other treatments was 0.5. The P.M./C.S. ratio was between 2.5 to 2.8 in all the treatments except in Stage II in which it was between 5.0 and 6.5. The material and water, in predeter-

mined ratios, were mixed together in a concrete mixer. The substrate samples were collected before it was loaded into the reactors.

Table 5
Waste Characteristics

Material	Stage	T.S. % w.b.	V . S .	T.K.N.	T.O.C.
Poultry	I	38.3	55.8	2.1	29.1
Manure	ΙI	49.9	55.4	1.3	27.6
	III	52.4	74.7	3.3	37.0
Corn	I	76.4	94.4	1.0	45.2
Stover	II	73.6	92.2	1.0	40.0
	III	84.4	93.6	1.0	41.5
High-solids	I-B	29.9	73.1	2.6	39.9
Fermenter	I-A	24.3	81.4	2.3	35.8
Residue	II-A2	25.3	52.3	2.6	29.6

The samples were analyzed for initial total solids, volatile solids, total nitrogen and total organic carbon. The initial substrate conditions in each treatment are reported in Table 6. The actual substrate conditions were slightly different than the theoretically determined conditions, which may be because of difficulty in getting repre-

sentative samples and/or inability to get homogeneous mix-ing.

Intital pH of the substrate in all the treatments of stage III, was between 6.5 and 8.5. No buffer chemical was added in order to observe the treatment performance in as simplified conditions as possible. However the studies on crop residues (11) indicated the need of buffer additions as much as 0.2 to 0.3 times the total feed quantity.

The biodegradability of the substrate was computed by taking the weighted average of the biodegradability of poultry manure and corn stover. The biodegradability of poultry manure and corn stover was found to be 0.87 and 0.65 respectively (13). The substrate containing X lb of poultry manure and Y lb of corn stover would have the biodegradability value:

The biodegradability of the substrate was nearly 0.8 for all treatments.

Table 6

Initial Substrate Conditions

Treatment	Quantity 1b	T.S. % w.b.			T.O.C.	C/N	рН
			12			<u> </u>	
I-A,I-B	13.8	34.0	76.6	1.2	36.0	.30.1	
II-A	28.9	27.8	63.7	1.1	30.7	28.1	;
II-B	30.2	26.4	70.2	1.1	34.8	31.5	
II-C	27.6	28.6	70.8	1.2	31.2	25.9	· · · · · · · · · · · · · · · · · · ·
III-A	18.2	30.0	86.8	2.3	37.7	17.2	7.2
III-B	13.0	34.4	82.8	2 • 2	38.7	17.7	7 . 4
III-C	15.3	35.2	87.8	2.3	38.5	17.3	7.8
III-D	14.5	35.2	85.2	2.1	38.9	18.0	8.0
III-E	15.6	35.1	78.8	2.2	37.5	17.1	7.8

5.2 GAS PRODUCTION AND ANALYSIS

Gas produced from each reactor was collected and measured individually by the water displacement method. Among the three replicates in the treatment III-A, III-B, III-C and III-E the one which showed the best performance, in terms of bio-gas production, was used to represent the particular treatment. In case of treatment III-D, both reactors III-D1

and III-D2 showed similar performance during the first three weeks, but reactor III-D1 is used to represent this treatment as it functioned much better than III-D2 in the later period.

Bio-gas and methane production rates were corrected to STP conditions (Temp.= 273° K and Press.= 1 atmp.) using Equation 8. The ambient temperature was between 22° C and 26° C (295° K to 299° K) while the pressure within the gas collector was very close to atmospheric pressure. An average factor of 0.9 was used to convert the gas volumes to STP conditions.

The daily gas production data of the selected digesters are presented in Appendix A. Appendix B shows the summary of the gas production data of all digesters.

Figure 10 and Figure 11 show the average bio-gas production rate in 1/day and accumulated bio-gas produced in liters of the five reactors in stage III for 54 days. During the first week a very high rate of gas production was observed from all the operating reactors. The accumulated gas produced in first ten days from reactor III-D1 was 46.7 liters, which was the maximum, while the minimum was from the reactor III-B3, 27.2 liters. On the seventh day, the bio-gas from selected digesters was first analyzed for methane content. The gas from all the reactors had less than 10% methane, which indicated very low energy produc-

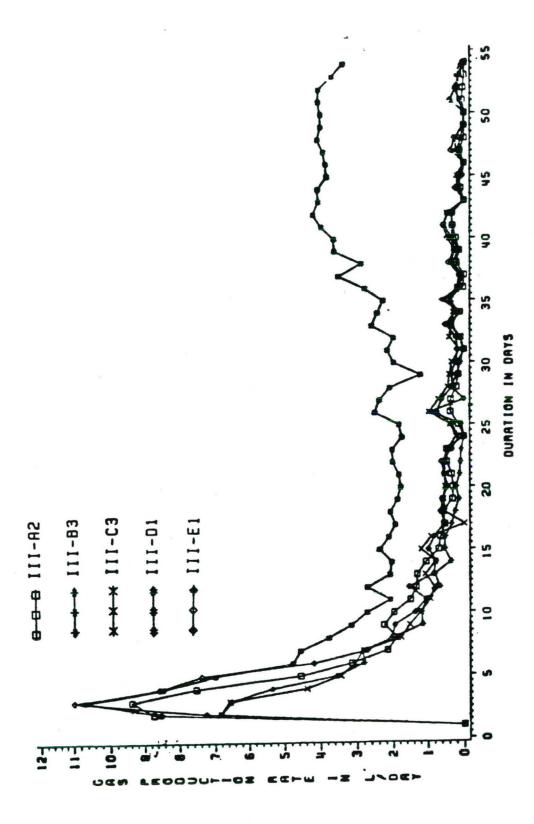


Figure 10: Average Gas Production Rate in Stage III

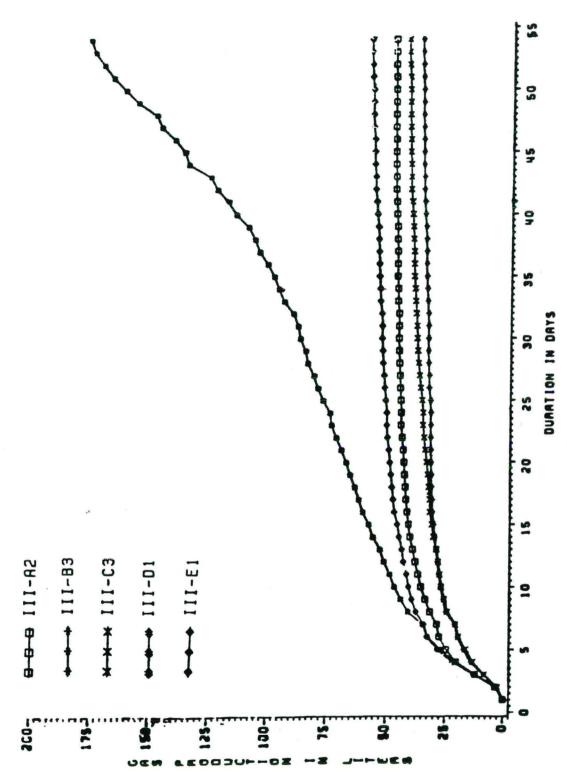


Figure 11: Accumulated Gas Production in Stage III

tion in the starting period. Methane analysis was done once per week.

The gas production rate of all the reactors declined sharply during the second and third week. However the methane content of the gas from all reactors except III-B3 went up. After 30 days, the gas production rate of III-D1 started increasing and went up to about 4.0 1/day on the 42nd day. Other reactors produced gas at almost a constant rate, about 0.5 1/day after 25 days. Although more than 45% of methane was observed from all the reactors except III-B3, very little gas was collected from III-A2, III-C3 and III-E1 after 45 days which indicated some leakage and/or some type of inhibition in the system. Despite resealing the lids of those reactors, none of them showed any improvement, indicating inhibition.

After 54 days only III-D1 reactor was observed for gas production and methane content. The gas production rate and accumulated gas produced from reactor III-D1 for 100 days are shown in Figure 12 and Figure 13. This reactor when terminated on the 100th day, was still producing gas at the rate of 2.3 1/day with a methane content of about 60%.

A similar pattern of gas production was observed from reactor I-A, as shown in Figure 14 and Figure 15. In the beginning, due to leakage, no gas was collected from this

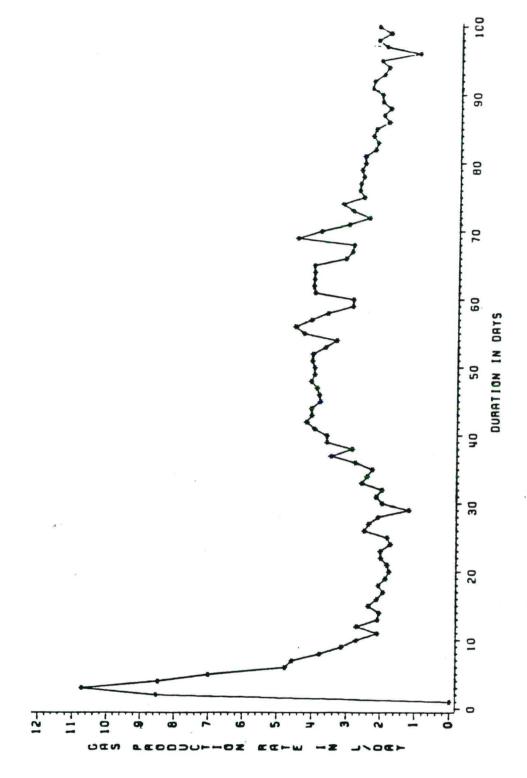


Figure 12: Average Gas Production Rate of Reactor III-D1

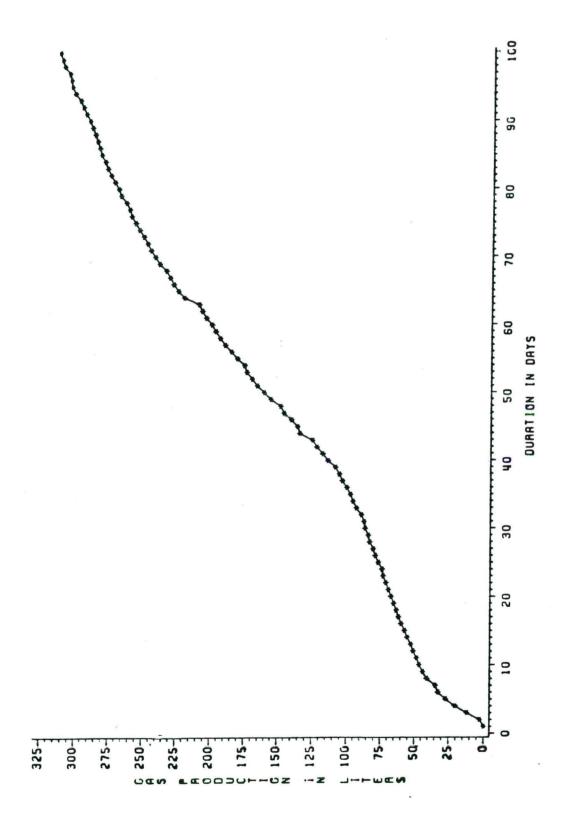


Figure 13: Accumulated Gas Production of Reactor III-D1

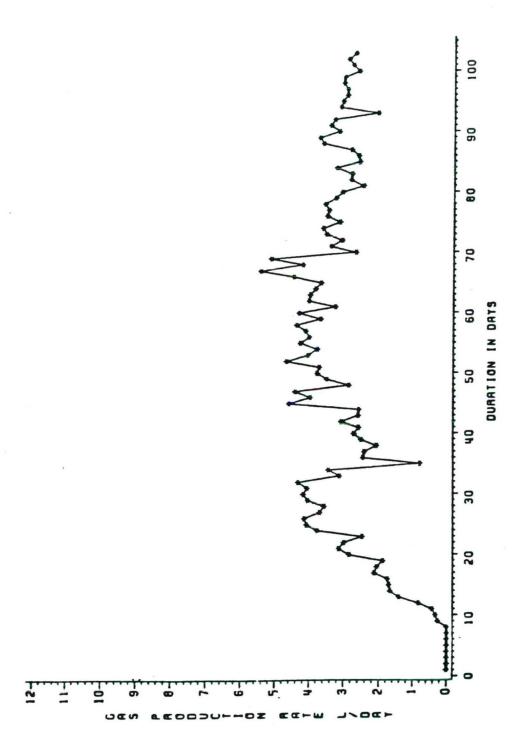


Figure 14: Average Gas Production Rate of Reactor I-A

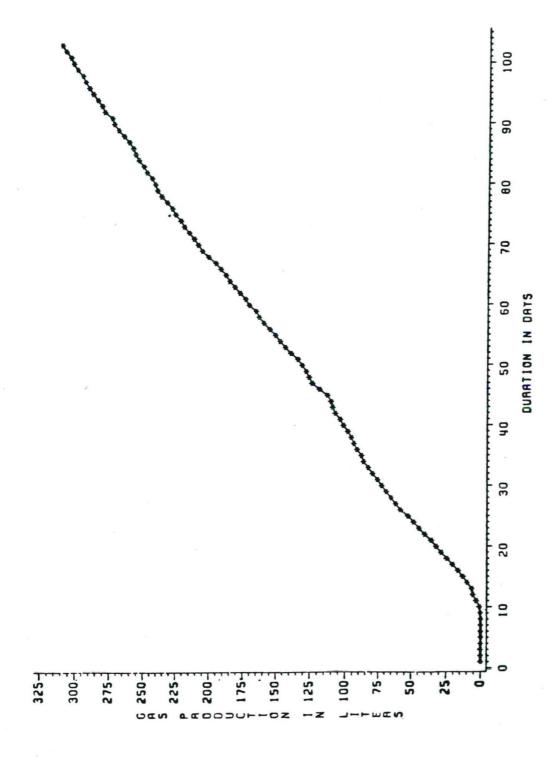


Figure 15: Accumulated Gas Production of Reactor I-A

reactor. After transfering the substrate into another digester, a gas production rate as high as 5.4 1/day was observed. This reactor was producing gas at the rate of 2.7 1/day, on the 118th day, when it was terminated. The substrate in this treatment had been inoculated with anaerobically digested municipal sludge. As the bio-gas analysis was not performed, the methane content of the gas was not determined. However the bio-gas produced after 20 days did burn, giving a blue flame, indicating the presence of methane.

Figure 16 and Figure 17 shows the performance of reactor II-A2, which was the only reactor operated successfully in that stage. The substrate in this treatment was inoculated with the residue from the high-solids fermenter I-B, which produced 46.8 liters of bio-gas in 46 days when it was terminated. Despite poor starting performance, the gas production rate of II-A2 was observed to increase from 1.0 l/day on the 30th day to 3.5 l/day on the 69th day. This reactor produced a total 129 liters of bio-gas in 7l days. The reactor was then terminated and residue was used to inoculate the substrate of the treatment III-E.

The bio-gas production rate of the reactor I-A, II-A2 and III-D1 showed the effect of volatile solids density in the digesters. I-A had a volatile solids density of 6.8 lb/ft 3 while II-A2 and III-D1 were loaded at the rate

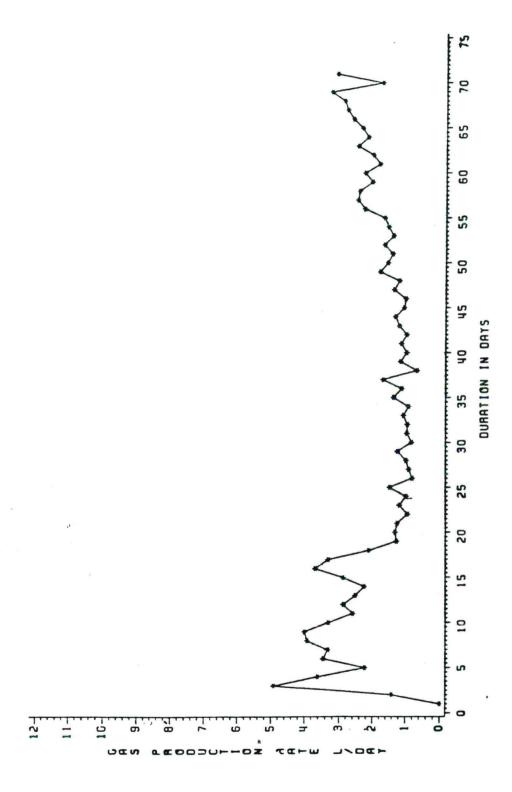


Figure 16: Average Gas Production Rate of Reactor II-A2

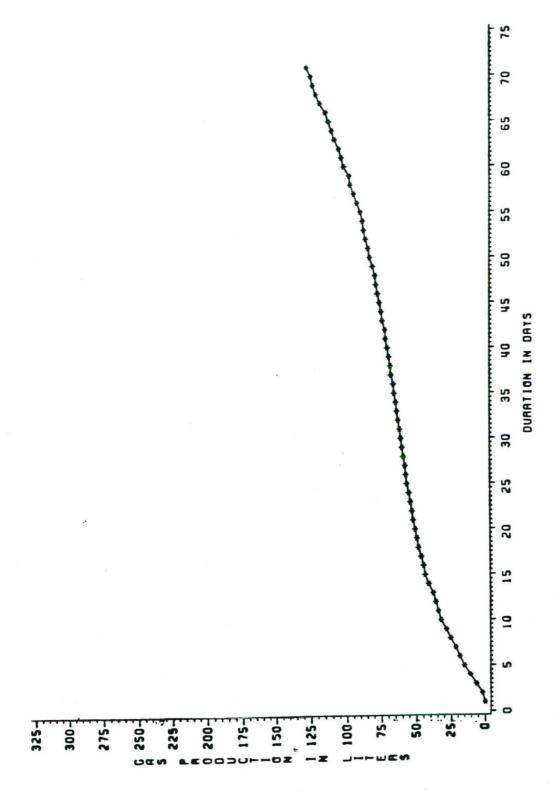


Figure 17: Accumulated Gas Production of Reactor II-A2

of 9.6 1b VS/ft^3 and 8.1 1b VS/ft^3 respectively. Reactor I-A and III-D1 were producing bio-gas at a higher rate in the first 70 days than II-A2. This indicated that the optimum volatile solids density in the digester might be between seven to eight 1b VS/ft^3 in order to achieve higher bio-chemical reaction rates.

5.2.1 Methane Production

The bio-gas from the functioning reactors in stage III was analyzed for methane content once a week. Regression analysis was done on the seven day interval methane content data of each reactor in order to get the equation which can approximate the daily methane content of the bio-gas. A third order regression equation gave a statistically significant relationship between the weekly observed methane content and the day. The regression equation was obtained for each reactor. The total methane production from each of these reactors is shown in Appendix A. Figure 18 shows the third order regression lines of methane content of the biogas from the reactors for 54 days.

Among all the operating reactors, bio-gas with maximum methane content was produced from the reactor III-Dl. Figure 19 shows the methane content of the bio-gas produced from III-Dl for 100 days.

Low methane content of the bio-gas from reactor III-B3 and III-C3 indicated fewer anaerobic bacteria in the system

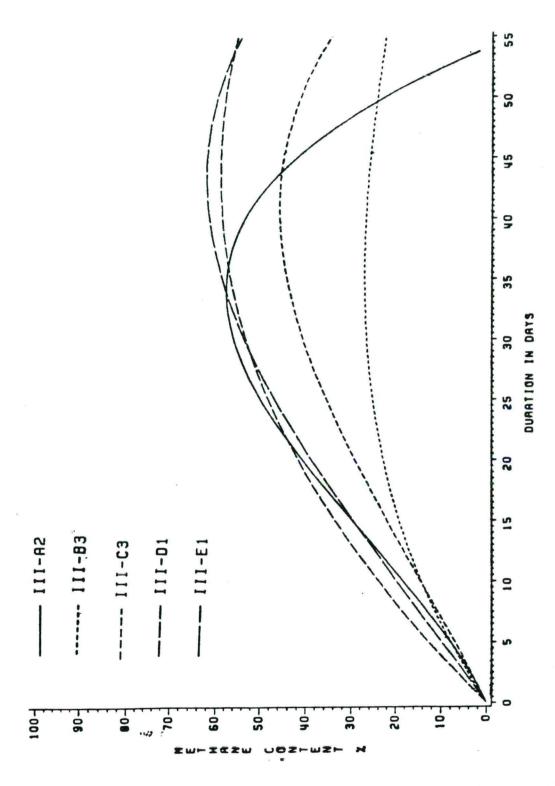
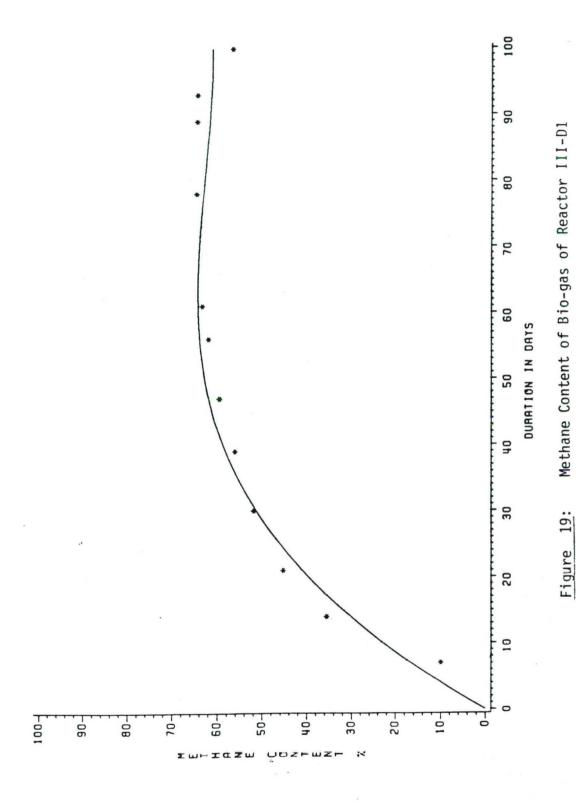


Figure 18: Methane Content of Bio-gas in Stage III



which might be due to poor seed and/or presence of oxygen. Reactor III-A2, III-D1, and III-El showed similar methane production for the first 35 days. The methane content of the bio-gas from III-A2 dropped sharply to less than 10% in next 15 days which indicated toxicity. The substrate of the reactor III-D1 and III-E1 was inoculated with the acclimated seed from the high-solids fermenter, containing anaerobic bacteria which resulted into faster reproduction of the bacteria in the system. The rapid growth of anaerobes could have prevented the acid accumulation in Although reactor III-El produced bio-gas at the rate and methane content comparable to III-D1 in the initial period, after 54 days much less gas was collected from III-El. The methane content of the bio-gas was about 59%. Reactor III-A2 started very well and produced bio-gas with about 55% after 30 days. The methane content then suddenly dropped to less than 10% in the next 15 days. The reason was not clearly understood for such a rapid drop in methane content, but it could be due to volatile acid accumulation and/or high concentration of soluble sulfur in the reactor.

5.2.2 Head-Space Gas Analysis

The head-space gas of all the reactors except III-Dl was checked, after sixty days, for the presence of ammonia and hydrogen sulfide gas using DRAGER tubes. The ammonia level in all the reactors was less than five ppm as NH₃ gas.

But hydrogen sulfide levels were observed to be greater than 200 ppm as ${\rm H_2S}$ gas. This was observed in all the reactors except III-El which showed a ${\rm H_2S}$ concentration of 120 ppm. As it was possible to measure the ${\rm H_2S}$ concentrations only up to 200 ppm with the DRAGER tubes, the exact concentration of hydrogen sulfide was undetermined. High hydrogen sulfide concentration in the bio-gas corresponds to the presence of soluble sulfur in the substrate. This could have inhibited the methanogens because of the activity of sulfate reducing bacteria competing for hydrogen with the methanogens.

After 100 days, when III-D1 was terminated, the ammonia and hydrogen sulfide concentration were measured in the head-space gas. Ammonia level was less than five ppm as NH_3 gas while hydrogen sulfide concentration was about 100 ppm as H_2S . The literature showed that in the presence of sulfur compounds, both the methane production and sulfate reduction can occur simultaneously in the presence of excess hydrogen. The presence of excess hydrogen may explain the reason for better performance of the reactor III-D1 than the others.

5.3 RESIDUE ANALYSIS

The residue from the reactors I-A, I-B and II-A2 was analyzed before it was used as an inoculum. The results of these analyses are reported in Table 5. All five reactors in stage III were terminated after 100 days. The residue samples were collected from the top, middle, and bottom of these reactors. A definite moisture gradient of 3% to 5%, lower at the top and higher at the bottom, was observed in all reactors.

The residue was analyzed for total solids, volatile solids, total nitrogen, ammonia nitrogen, total organic carbon The results of these analyses are presented in and pH. Table 7. The pH analyses of the residue from each treatment indicated lower pH values than the initial substrate. However the pH of the residue from the reactor III-Dl still 7.5, the residue from the rest of the reactors showed pH between 6.0 and 6.7. This indicated some acidity problems in the unsuccessful reactors. The volatile acids accumulation in those reactors would have inhibited the activity of methanogenic bacteria. Mixing the substrate with buffer chemicals like ammonium bicarbonate, sodium bicarbonate or calcium bicarbonate at the begining could have helped to neutralize the excess acids preventing toxicity. The samples prepared for pH analysis were used for COD analysis after appropriate dilution. Procedure #508 of

Standard Methods (25) was followed for this analysis. The residue from reactor III-Dl had the lowest COD value of 8,560~mg/l (Appendix C).

Table 7

Residue Analysis

Reactor	T.S. % w.b.	V . S .		T.O.C.	V.S.D. % I.V.S.	рН
III-A2	24.8	81.8	2 • 4	38.7	21.3	6.1
III-B3	29.0	75.0	2.6	37.7	24.3	6.7
III-C3	30.6	75.0	2.7	38.3	25.5	6.2
III-D1	30.8	75.6	2.4	38.6	23.2	7.5
III-E1	30.6	73.3	2.4	38.9	18.6	6.4

The moisture content at the end of each treatment had increased from the initial moisture content (Table 7). The alteration of the water-binding characteristics of the substrate during the digestion process as well as some loss of total solids from the system may explain the increase in the moisture content. The initial total solids in five treatments of stage III were between 30% to 35% while that of the residue was between 25% to 30%.

The digester III-Al, III-B3, III-C3 and III-E1 showed volatile solids reduction between 0.8 lb to 1.2 lb. The bio-gas collected from these reactors was much less than that from the reactor III-D1, which showed a volatile solids reduction of 1.0 lb. The rate of volatile solids reduction was much slower than what is observed in slurry type digestion. However it was comparable to Jewell's work on crop residue.

Initial biodegradable volatile solids in reactor III-D1 were 3.4 lb. By linear extrapolation of the volatile solids reduction in the 100 day test of III-D1, it was estimated that about 310 days would be require for 90% reduction in the biodegradable volatile solids. If the reactor was operated for this period without any inhibition, it would produce 17.7 ft of methane which is about 17,000 BTUs in 310 days. Although the methane production per 1b volatile solids destroyed is comparable to the slurry type anaerobic digestion, the rate of methane production in high-solids anaerobic digestion is very slow.

The total volatile solids reduction in the treatments did not differ greatly which indicated no appreciable effects of initial total solids in the range of 30% to 35% on biodegradation of the substrate. However due to certain unavoidable problems, leakage and toxicity, it was not possible to determine the influence of initial total solids

concentration on rate and quality of bio-gas production. The acclimated seed from the previously operating high-solids fermenter did show some advantages over the municipal sludge to initiate the digestion process.

Total nitrogen content in all the treatments was not change during the process. However the ammonia to total nitrogen ratio of the residue was observed to be between 0.6 and 0.9. This change indicated part of organic nitrogen converted to ammonia nitrogen. Total organic carbon was reduced by 10.0% to 19.1% during the digestion process.

Chapter VI

SUMMARY

With the trend in the poultry industry towards growing a large number of birds at one place, manure disposal has become a critical issue from the pollution control point of view. Although the poultry manure is a good source of organic fertilizer as well as bio-energy, poorly managed manure handling can make it a major source of environmental pollution. Anaerobic digestion of poultry manure not only stabilizes the manure and makes it less of a pollutant but also produces bio-gas containing methane, which is a source of energy.

In large facilities holding thousand of birds the manure is usually collected once or twice a year. The manure collected from such facilities needs to be treated in a batch rather than on a continuous basis. Conventional slurry-type anaerobic digestion requires large quantities of water for dilution and the digestion process is usually a continuous type which needs daily manure loading. The process of treating poultry manure in a batch system at higher solid contents appears to have more potential than the conven-

tional process. Treatment at higher solids concentration would not only increase the organic matter loading rate but also reduce the amount of heat required to maintain temperature inside the reactor.

Twenty liter (0.7 ft³) capacity digesters were started with a mixture of poultry manure, corn stover and inoculum at the initial total solids content between 30% and 35% in stage III. The substrate in three treatments was inoculated with municipal sludge while in two other treatments acclimated seed from a high-solids fermenter was used for inoculation. The volatile solids density in the digesters were 7.0 lb/ft³ to 8.8 lb/ft³. After 54 days of operation only one reactor was operating. The leakage during the initial period might have caused an imbalance between the process of acidogenesis and methanogenesis of the organic matter, causing volatile acid accumulation, a probable cause of inhibition.

The reactor III-D1, which did not face any problems, was operated for 100 days. The substrate of this reactor was inoculated with an acclimated seed from a high-solids fermenter which was operated for 118 days. The maximum methane content of the bio-gas from this reactor was 65%. It was observed to produce gas at a rate of 10.7 1/day on the second day, then dropped sharply to 2.1 1/day by the eleventh day of operation. The gas production rate continued to drop

up to the 29th day when it produced 1.2 1/day. For the next 15 days a gradual increase in gas production was observed. The methane content by that time reached 60%. From the 45th day to the 65th day, reactor produced gas at the rate of 3.9 1/day to 4.1 1/day. After 65 days gas production gradually dropped to 2.3 1/day with a constant methane content of 62% on the 100th day when it was terminated.

Accumulated bio-gas produced in 100 days was 313.1 litres (11.1 ft³), while total methane produced was 163.6 litres (5.8 ft³). The final substrate analyses showed a reduction of 1.0 lb volatile solids which is 23.2% of the initial volatile solids. The solids content of the substrate was reduced to 30.8% from initial value of 35.2% w.b. The bio-gas production per pound of volatile solids destroyed was 11.1 ft³, which is comparable to the values 9 to 11 ft³/1b VSD in a slurry type process (3). However the retention time in high-solids fermentation was 100 days, in slurry type process it is 30 to 52 days. The methane produced per 1b volatile solid destroyed was 5.8 ft³, which is also comparable to the conventional slurry type process.

The reactors which stopped producing any significant amount of gas after 54 days showed higher hydrogen sulfide content in the head space gas than the reactor III-D1. The pH of the residue from those reactors was between 6.1 and

6.7 while that of the residue from the reactor III-Dl was 7.5. These analyses indicated high sulfur and volatile acid levels in the reactor III-A2, III-B3, III-C3 and III-E1 which might have inhibited the microbial activities in those reactors.

The volatile solids reduction between 18.6% to 25.5% of the initial volatile solids was observed in all the treatments which indicated very little effects of total solids content in the range of 30% to 35% on biodegradation of organic matters.

The digester I-A and II-A2 were operated successfully in stage I and stage II respectively. The substrate of I-A was inoculated with municipal sludge while that of II-A2 was inoculated with the residue from a high-solids fermenter which was operated for 47 days. I-A produced 311.9 liters (11.0 ft³) bio-gas in 118 days and showed 1.1 lb of volatile solids reduction, 25.6% of the initial volatile solids. II-A2 produced 129.0 liters (4.6 ft³) bio-gas in 71 days and 0.7 lb volatile solids were destroyed, 25.0% of the initial volatile solids. The methane content of the bio-gas in these experiments was not determined.

The bio-gas production rates from I-A, II-A2 and III-D1 in terms of liters/lb VSD per day was 2.4, 2.6 and 3.1 respectively. These values indicated that the type of seeding material does have an impact on the digestion process.

The acclimated seed from a successfully operated high-solids fermenter gave better inoculation of the substrate and higher bio-gas production rate than the municipal sludge.

Chapter VII

CONCLUSION

- High-solids anaerobic fermentation of the poultry manure mixed with corn stover as a source of organic carbon at 30% - 35% initial total solids produced biogas quantitatively comparable to the slurry type anaerobic fermentation.
- 2. Gas production per pound volatile solids reduction from the digester containing substrate inoculated with the residue from a high-solids anaerobic fermenter was higher than the one containing substrate inoculated with municipal sludge. A seed to feed ratio of 0.05 on a wet basis in stage II gave satisfactory gas production.
- 3. The digestion process was monitored for 100 days. Volatile solids reduction of 23% to 25% occured. Based on this fact a retantion time of 180 days is needed for 50% volatile solids reduction, which would give satisfactory stabilization.
- 4. The bio-gas production per pound of volatile solids reduced was 11.1 cubic foot, which is comparable to

values of nine to eleven cubic foot in a slurry type digestion. The methane production per pound volatile solids reduced was 5.8 cubic foot, which is also comparable to the conventional slurry type digestion.

5. Toxicity was observed in several digesters as evidenced by low gas production and low methane content of the gas. These digesters had a low pH, 6.2 to 6.7, which indicates a volatile acids accumulation. Also, hydrogen sulfide in the head space gas was measured over 200 ppm which indicates a sulfide toxicity.

Chapter VIII

FUTURE RESEARCH RECOMMENDATIONS

- 1. Evaluate the effects of
 - er, between five and ten pound volatile solids per cubic foot, at solids content 30% to 35%
 - b. chemical pre-treatment of the substrate, mainly buffer addition
 - c. recirculation of a part of bio-gas in the digester
 - on the overall performance of the system.
- 2. Use reactors with perforated bottom in order to maintain uniform moisture content within the reactor.
- 3. Modify the exiting procedures to analyze the waste at high solids for chemical oxygen demand, volatile acids and soluble sulfur concentration.
- 4. Collect data on CODs of the substrate before and after the digestion in order to determine the stability of the waste material.
- 5. Determine the possibilities of using aerobic fermentation at start-up to raise the substrate temperature.

Operate a bench scale high-solids fermenter for a longer period, at least six months, and determine the net energy production.

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Appendix A

DAILY GAS PRODUCTION AND METHANE CONTENT

	DIG	ESTER=I-A		
DAY	GAS PROD.RATE	(1/D)	ACC.GAS	PROD. (1)
1	0.0			0.0
2	0.0			0.0
3	0.0			0.0
4 5	0.0 0.0			0.0
6	0.0			0.0 0.0
7	0.0			0.0
8	0.0			0.0
9	0.3			0.3
10				0.3
11	0.3			0.9
12	*			0.9
13	:			0.9
14 15	:			0.9
16	0.4			3.0
17	*			3.0
18				3.0
19	0.8			5.5
20	1_4			6.5
21				6.5
22 23	1.7			0.0
24	* 1.7			0.0
25	*			3.1
26	1.8			6.6
27				6.6
28	2.1			0.6
29	*		2	0.6
30	2. 1			4.6
31	.*.			4.6
32 33	1.9 2.8			8.6
34	3. 1			2.1 5.9
35	*			5.9
36	3.0			0.4
37	2.5			4_4
38	3.8			8.4
39	4.1			2.4
40	4.2 3.7			8.3
42	3.7			2.0
43	4.1			5.5 9.3
44	4-2			2.7
45	4.1			6.1
46	4.3			9.9
47	3.1			3.3
48	3.5			5.9
49 50	*			5.9
51	0.8 2.5			8.5
52	2.4			1.9
53	2. 1			5.5
54	2.5			3.9

DAY GAS PROD.RATE (1/D) ACC.GAS PROD. (1) 55 2.7 102.3 56 2.6 104.5 57 3.1 108.0 58 2.6 109.9 59 2.6 111.2 60 4.6 113.7 61 4.0 119.3 62 4.4 124.4 63 2.9 126.6 64 3.5 128.5 65 3.8 131.6 66 3.7 134.8 67 4.7 139.8 68 4.1 144.0 69 3.8 147.8 70 4.3 151.5 71 4.0 155.9 72 4.1 160.2 73 4.4 164.0 74 3.7 166.2 75 4.3 171.4 76 3.3 174.2 77 4.0 178.2 79
56 2.6 104.5 57 3.1 108.0 58 2.6 109.9 59 2.6 111.2 60 4.6 113.7 61 4.0 119.3 62 4.4 124.4 63 2.9 126.6 64 3.5 128.5 65 3.8 131.6 66 3.7 134.8 67 4.7 139.8 68 4.1 144.0 69 3.8 147.8 70 4.3 151.5 71 4.0 155.9 72 4.1 160.2 73 4.4 164.0 74 3.7 166.2 75 4.3 171.4 76 3.3 174.2 77 4.0 178.2 80 3.7 188.8 81 4.5 192.8 82 5.4 196.8 83 4.2 202.0 84
56 2.6 104.5 57 3.1 108.0 58 2.6 109.9 59 2.6 111.2 60 4.6 113.7 61 4.0 119.3 62 4.4 124.4 63 2.9 126.6 64 3.5 128.5 65 3.8 131.6 66 3.7 134.8 67 4.7 139.8 68 4.1 144.0 69 3.8 147.8 70 4.3 151.5 71 4.0 155.9 72 4.1 160.2 73 4.4 164.0 74 3.7 166.2 75 4.3 171.4 76 3.3 174.2 77 4.0 178.2 80 3.7 188.8 81 4.5 192.8 82 5.4 196.8 83 4.2 202.0 84
57 3. 1 108.0 58 2.6 109.9 59 2.6 111.2 60 4.6 113.7 61 4.0 119.3 62 4.4 124.4 63 2.9 126.6 64 3.5 128.5 65 3.8 131.6 66 3.7 134.8 67 4.7 139.8 68 4.1 144.0 69 3.8 147.8 70 4.3 151.5 71 4.0 155.9 72 4.1 160.2 73 4.4 164.0 74 3.7 166.2 75 4.3 171.4 76 3.3 174.2 77 4.0 178.2 78 4.0 182.2 79 3.8 186.2 80 3.7 188.8 81 4.5 192.8 82 5.4 196.8 83
59 2.6 111.2 60 4.6 113.7 61 4.0 119.3 62 4.4 124.4 63 2.9 126.6 64 3.5 128.5 65 3.8 131.6 66 3.7 134.8 67 4.7 139.8 68 4.1 144.0 69 3.8 147.8 70 4.3 151.5 71 4.0 155.9 72 4.1 160.2 73 4.4 164.0 74 3.7 166.2 75 4.3 171.4 76 3.3 174.2 77 4.0 178.2 78 4.0 182.2 79 3.8 186.2 80 3.7 188.8 81 4.5 192.8 82 5.4 196.8 83 4.2 202.0 84 5.1 206.8
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64 3.5 128.5 65 3.8 131.6 66 3.7 134.8 67 4.7 139.8 68 4.1 144.0 69 3.8 147.8 70 4.3 151.5 71 4.0 155.9 72 4.1 160.2 73 4.4 164.0 74 3.7 166.2 75 4.3 171.4 76 3.3 171.4 77 4.0 178.2 78 4.0 182.2 79 3.8 186.2 80 3.7 188.8 81 4.5 192.8 82 5.4 196.8 83 4.2 202.0 84 5.1 206.8
65 3.8 131.6 66 3.7 134.8 67 4.7 139.8 68 4.1 144.0 69 3.8 147.8 70 4.3 151.5 71 4.0 155.9 72 4.1 160.2 73 4.4 166.2 75 4.3 171.4 76 3.3 171.4 76 3.3 171.4 76 3.8 174.2 77 4.0 178.2 78 4.0 182.2 79 3.8 186.2 80 3.7 188.8 81 4.5 192.8 82 5.4 196.8 83 4.2 202.0 84 5.1 206.8
66 3.7 134.8 67 4.7 139.8 68 4.1 144.0 69 3.8 147.8 70 4.3 151.5 71 4.0 155.9 72 4.1 160.2 73 4.4 166.2 75 4.3 171.4 76 3.7 166.2 77 4.0 178.2 78 4.0 178.2 79 3.8 186.2 79 3.8 186.2 80 3.7 188.8 81 4.5 192.8 82 5.4 196.8 83 4.2 202.0 84 5.1 206.8
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72 4.1 160.2 73 4.4 166.2 74 3.7 166.2 75 4.3 171.4 76 3.3 174.2 77 4.0 178.2 78 4.0 182.2 79 3.8 186.2 80 3.7 188.8 81 4.5 192.8 82 5.4 196.8 83 4.2 202.0 84 5.1 206.8
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74 3.7 166.2 75 4.3 171.4 76 3.3 174.2 77 4.0 178.2 78 4.0 182.2 79 3.8 186.2 80 3.7 188.8 81 4.5 192.8 82 5.4 196.8 83 4.2 202.0 84 5.1 206.8
76 3.3 174.2 77 4.0 178.2 78 4.0 182.2 79 3.8 186.2 80 3.7 188.8 81 4.5 192.8 82 5.4 196.8 83 4.2 202.0 84 5.1 206.8
77 4.0 178.2 78 4.0 182.2 79 3.8 186.2 80 3.7 188.8 81 4.5 192.8 82 5.4 196.8 83 4.2 202.0 84 5.1 206.8
78
79 3.8 186.2 80 3.7 188.8 81 4.5 192.8 82 5.4 196.8 83 4.2 202.0 84 5.1 206.8
80 3-7 188-8 81 4-5 192-8 82 5-4 196-8 83 4-2 202-0 84 5-1 206-8
81 4.5 192.8 82 5.4 196.8 83 4.2 202.0 84 5.1 206.8
82 5.4 196.8 83 4.2 202.0 84 5.1 206.8
83 4.2 202.0 84 5.1 206.8
84 5.1 206.8
86 3.4 212.7
87 3.1 216.4
88 3.5 220.2
89 3.6 222.7
90 3.2 226.7
91 3.5 229.3
92 3.5 233.3
93 3.6 237.6
94 3.3 240.8
95 3.1 242.6
96 2.5 245.1 97 2.9 249.1
2,7,1
98 2.8 251.3 99 3.3 255.4
100 2.6 257.9
101 2.6 259.5
102 2.8 262.4
103 3.6 266.5
104 3.7 270.6
105 3.2 273.8
106 3.4 275.3
107 3.3 280.9
108 2-1 282-8

	DIG	ESTER=I-A			
DAY	GAS PROD. RATE	(1/D)	ACC.GAS	PROD. (1)	
109	3.1		28	36.0	
110	3.1		289-4		
111	3.0		292.1		
112	3.0		294.9		
113	3.1		296.9		
114	3. 1		301.1		
115	2.6		30	3.7	
116	2.8		30	5.5	
117	2.9		30	9.2	
118	2.7		3	11.9	

^{* -} Data not available

	DIGESTE	R=II-A2
DAY	GAS PROD.RATE (1/	D) ACC.GAS PROD. (1)
1	0.0	0.0
2	1_4	2.0
3	4.9	6.0
4	3.6	10.5
5	2.2	15.0
6	3.5 3.3	18.3 21.5
7 8	3.9	25. 1
9	4.0	28.2
10	3.3	32. 2
11	2.6	34.1
12	2.9	36. 1
13	2.5	38.1
14	2.3	41.2
15	2.9	43.6
16	3.7	44.6
17	3.3	46.4
18	2.1	48.3
19	1.3	49.3
20	1.3	50.8 52.1
21	1.2 0.9	53. 1
22 23	1. 2	54.2
24	1.0	55. 3
25	1.5	56.5
26	.0.8	57.4
27	0.9	58.1
28	1.0	59. 1
29	1. 2	60.3
30	0.8	61.1
31	1_0	62.1
32	1.0	63.1
3 3	1.1	63.8 64.7
34	0.9	65.8
35 36	1. 1	66.5
37	1. 7	68.0
38	0.7	68.7
39	1. 2	69.8
40	1.0	70.8
41	1. 1	72.0
42	1.0	72.5
43	1. 2	74.3
44	1.3	75.3
45	1.1	76.5
46	1.0	77.7 79.0
47 48	1.4 1.2	79.0 79.7
48	1.8	81.5
50	1.6	83.5
51	1. 4	84.6
52	1.7	86.7
53	1. 4	88.0
54	1.5	88.9

	DIGESTE	R=II-A2
DAY	GAS PROD.RATE (1/	D) ACC.GAS PROD. (1)
5 5	1.7	90.6
56	2.3	92.9
57	2.5	95.6
58	2.4	98.1
59	2.1	99.0
60	2.3	102.8
61	1.8	104.5
62	2.0	106.5
63	2.5	109.3
64	2.2	111.3
65	2.4	113.5
66	2.6	115.5
67	2.8	119.5
68	2.9	122.3
69	3.3	124.5
70	1.7	125.9
7 1	3.1	129.0

		DIGESTER=	III-12	
DAY	RATE (1/D)	TOTAL (1)	CH4 (1/D)	TOTAL CH4 (1)
1	0.0	0.0	0.0	0.0
2	8.7	3.3	0.2	0_1
3	9.4	12.2	0-4	0.5
4	7.6	20.3	0.5	1.0
5	4.6	24.5	0.4	1.3
6	3.1	27.6	0.3	1.6
7	2.2	28.6	0.3	1.8
8	1.9	31.7	0.3	2. 2
9	2.3	33.7	0.4	2.5
10	2.0	35.6	0.4	2.9
11	1.5	36.9	0.3	3.1
12	1.3	38.1	0.3	3.4
13	1.3	39.2	0.3	3.7
14	1.0	40-4	0.3	4.0
15	0.7	40.9	0.2	4.2
16	0.6	41.7	0.2	4_4
17	0.5	42.2	0.2	4-6
18	0.6	42.6	0.2	4.7
19	0.3	42.9	0_1	4.8
20	0.3	43.2	0_1	5.0 5.1
21	0.3	43.5	0.1	5.3
22	0.5	44-0	0.2	5.5
23	0.5	44.5 44.5	0.2	5.5
24	0.0	44.7	0.1	5.6
25 26	0.4	45.0	0. 2	5.8
27	0.4	45.2	0.2	5.9
28	0.2	45.5	0.1	6.1
29	0.2	45.6	0.1	6.2
30	0.2	45.8	0. 1	6.2
31	0.0	45.8	0.0	6.2
32	0.1	45.9	0. 1	6.3
33	0.3	46.3	0.2	6.5
34	0.1	46.4	0.1	6.6
35	0.4	46.7	0.2	6.7
36	0.0	46.7	0.0	6.8
37	0.0	46.7	0.0	6.8
38	0.2	46.8	0. 1	6-8
39	0.1	46.9	0-1	6.9
40	0.2	47.2	0.1	7.0
41	0.3	47.4	0.2	7.1 7.3
42	0.3	47.7 47.7	0.2 0.0	7.3
43	0_0	47.8	0.0	7.3
44	0-1	47.9	0-1	7.4
45		47.9	0.0	7.4
46	0_0 0_1	48-0	0-0	7.4
48	0.0	48.0	0_0	7.4
49	0.0	48.0	0.0	7.4
50	0.0	48.0	0.0	7.4
51	0.1	48.0	0.0	7.4
52	0.0	48.1	0.0	7.4
53	0.0	48.1	0.0	7.4
54	0.0	48.1	0.0	7.4

		DIGESTER=	III-B3	
DAY	RATE (1/D)	TOTAL (1)	CH4 (1/D)	TOTAL CH4 (1)
1	0.0	0.0	0.0	0.0
2	6.8	2.8	0.2	0_1
3	6.5	8-4	0.3	0-4
4	5.4	14-2	0.3	0.7
5	3.6	17.3	0.3	1.0
6	2.8	19.9	0.3	1.2
7	2.8	21.2	0.3	1.4
8	2.0	24.8	0.2	1.8
9	1.2	26.0 27.2	0.2 0.2	2.0 2.1
10	1.0	28.0	0.2	2.3
12	0.7	28.7	0.1	2.4
13	0.8	29.3	0.1	2.5
14	0.4	29.9	0.1	2.6
15	0.5	30.4	0.1	2.7
16	0.5	30.9	0.1	2.8
17	0.3	31.2	0.1	2.9
18	0.2	31.4	0. 1	2.9
19	0.1	31.6	0.0	2.9
20	0.2	31.7	0.0	3.0
21	0.1	31.9	0.0	3.0
22	0.1	32.0	0.0	3.0 3.0
23	0-1	32.1	0.0 0.0	3.0
24 25	0.0	32.1 32.1	0_0	3.1
26	0.9	32.8	0.2	3. 2
27	0.0	32.8	0.0	3.2
28	0.3	33.1	0.1	3.3
29	0.2	33.3	0.1	3.4
30	0.1	33.4	0.0	3.4
31	0.0	33.4	0.0	3.4
32	0_1	33.5	0.0	3.4
33	0-4	33.9	0.1	3.5
34	0.1	34.0	0.0	3.6 3.7
35 36	9.6 0.1	34.4 34.5	0.2 0.0	3.7
37	0.1	34.6	0.0	3.7
38	0.3	34.7	0-1	3.7
39	0.1	34.8	0.0	3.8
40	0.3	35.3	0.1	3.9
41	0.3	35.4	0.1	3.9
42	0-4	35.8	0.1	4.0
43	0.0	35.8	0.0	4.0
44	0.2	36.1	0.0	4.1
45	0.1	36.2	0.0	4.1
46	0.0	36.2 36.4	0.0 0.0	4.1
47	0.2 0.1	36.4	0.0	4.2
49	0.0	36.4	0.0	4.2
50	0.0	36-4	0.0	4.2
51	0.1	36.6	0.0	4.2
52	0.2	36.8	0.0	4.3
53	0.1	37.0	0.0	4.3
54	0.0	37.0	0.0	4.3

		DIGESTER=	III-c3	
DAY	RATE (1/D)	TOTAL (1)	CH4 (1/D)	TOTAL CH4 (1)
1	0.0	0.0	0.0	0.0
2	6.8	2.3	0.2	0.1
3	6.6	8.2	0.3	0.3
4	4 - 4	13.2	0.2	0.6
5 6	3.5	16.4	0.2	0_8
7	3.1	19.6	0.3	1.1
8	2.8 1.8	20.9 24.3	0.3	1.2
ğ	1.5	25.7	0.2 0.2	1.6
10	1.2	26.9	0.2	1.8 2.0
11	0.9	27.6	0.1	2.1
12	0.8	28.4	0.1	2. 2
13	1.1	29.2	0.2	2.4
14	0.8	30.3	0.2	2.6
15	1.2	31.2	0.3	2.8
16	0.9	32.0	0.2	3.0
17 18	0.0	32.0	0.0	3.0
19	0.5 0.6	32.5 33.0	0.1	3.1
20	0.5	33.5	0.2	3.2
21	0.5	34.1	0.2	3.4 3.6
22	0.5	34.6	0.2	3.7
23	0.5	35.1	0_1	3.9
24	0.2	35.2	0.1	3.9
25	0.4	35.7	0_1	4.1
26	1.0	36.3	0.4	4.3
27	0.7	36.8	0.3	4.5
28 29	0-4	37.3	0.2	4.7
30	0.4	37.7 38.1	0.2 0.1	4.9
31	0.1	38.1	0. 1	5.0 5.0
32	0.4	38.5	0. 2	5. 2
33	0.3	38.9	0.2	5.4
34	0.3	39.2	0. 1	5.5
35	0.3	39.4	0.2	5.6
36	0.2	39.6	0_ 1	5.7
37	0.1	39.7	0.1	5.7
38 39	0.3	39.9	0.2	5.8
40	0.3 0.3	40-1 40-6	0_1	5.9
41	0.5	40.8	0.2 0.2	6.1 6.2
42	0.5	41.3	0.2	6.5
43	0.0	41.3	0.0	6.5
4.4	0.2	41.5	0. 1	6.6
45	0.2	41.6	0.1	6.6
46	0.0	41.6	0.0	6.6
47	0.2	41.8	0.1	6.7
48	0-2	42.0	0.1	6.8
4 9	0.0	42.0	0.0	6.8
51	0-0 0-1	42.0 42.2	0.0	6.8
52	0.2	42.4	0.1 0.1	6.8 6.9
53	0.2	42.7	0.1	7.0
54	0.1	42.7	0.0	7.0
	PMC (200 AC)			

DAT RATE (1/D) TOTAL (1) CE4 (1/D) TOTAL CB4 (1) 1 0.0 0.0 0.0 0.0 2 8.5 2.9 0.4 0.1 3 10.7 12.0 0.8 0.8 4 8.5 20.7 0.8 1.7 5 7.0 27.2 0.9 2.5 6 4.8 32.4 0.7 3.2 7 4.6 34.5 0.8 3.6 8 3.8 40.8 0.7 4.8 9 3.2 44.0 0.7 5.4 10 2.7 46.7 0.6 6.5 11 2.1 48.7 0.5 6.5 12 2.7 51.1 0.7 7.2 14 2.1 55.6 0.6 8.5 15 2.4 57.6 0.8 9.1 16 2.1 60.1 0.7 10.0 17 <td< th=""><th></th><th></th><th> DIGESTER=</th><th>III-D1</th><th></th></td<>			DIGESTER=	III-D1	
2 8.5 2.9 0.4 0.1 3 10.7 12.0 0.8 0.8 4 8.5 20.7 0.8 1.7 5 7.0 27.2 0.9 2.5 6 4.8 32.4 0.7 3.2 7 4.6 34.5 0.8 3.6 8 3.8 40.8 0.7 4.8 9 3.2 44.0 0.7 5.4 10 2.7 46.7 0.6 6.0 11 2.1 48.7 0.5 6.5 11 2.1 48.7 0.5 6.5 12 2.7 51.1 0.7 7.2 13 2.1 52.9 0.6 7.7 14 2.1 55.6 0.6 8.5 15 2.4 57.6 0.8 9.1 16 2.1 60.1 0.7 10.0 17 1.9 62.0 0.7 10.7 18 2.1 63.7 0.8 9.1 19 1.9 65.6 0.7 12.0 20 1.8 67.4 0.7 12.7 21 1.8 69.4 0.8 13.5 22 2.0 71.4 0.9 14.4 23 2.0 73.4 0.9 14.4 23 2.0 73.4 0.9 14.4 23 2.0 73.4 0.9 15.3 24 1.7 77.0 0.8 17.0 25 1.8 77.0 0.8 17.0 26 2.5 79.2 1.2 18.8 27 2.4 80.8 1.2 18.8 28 2.1 83.4 1.0 20.1 29 1.2 84.4 0.6 20.6 30 2.0 86.5 1.0 20.1 31 2.2 87.5 1.1 22.2 32 2.0 89.5 1.1 23.2 33 2.6 93.3 1.4 25.3 34 2.5 95.7 1.4 20.6 20.6 35 2.3 97.7 1.3 27.7 36 2.8 100.5 1.6 22.3 37 3.5 104.0 2.0 31.3 38 2.9 106.0 1.7 32.5 39 3.6 108.8 2.1 34.1	DAY	BATE (1/D)	TOTAL (1)	CH4 (1/D)	TOTAL CH4 (1)
3 10.7 12.0 0.8 0.8 4 8.5 20.7 0.8 1.7 5 7.0 27.2 0.9 2.5 6 4.8 32.4 0.7 3.2 7 4.6 34.5 0.8 0.7 4.8 9 3.2 44.0 0.7 5.4 10 2.7 46.7 0.6 6.0 11 2.1 48.7 0.5 6.5 12 2.7 51.1 0.7 7.2 13 2.1 52.9 0.6 7.7 14 2.1 55.6 0.8 9.1 15 2.4 57.6 0.8 9.1 16 2.1 60.1 0.7 10.0 17 1.9 62.0 0.7 10.7 18 2.1 63.7 0.8 11.3 19 1.9 65.6 0.7 12.0 20 1.8 67.4 0.7 12.7 21 1.8 69.4 0	1	0.0	0.0	0.0	
4 8.5 20.7 0.8 1.7 5 7.0 27.2 0.9 2.5 6 4.8 32.4 0.7 3.2 7 4.6 34.5 0.8 3.6 8 3.8 40.8 0.7 4.8 9 3.2 44.0 0.7 5.4 10 2.7 46.7 0.6 6.0 11 2.1 48.7 0.5 6.5 12 2.7 51.1 0.7 7.2 13 2.1 52.9 0.6 7.7 14 2.1 55.6 0.6 8.5 15 2.4 57.6 0.8 9.1 16 2.1 60.1 0.7 10.7 18 2.1 63.7 0.8 9.1 17 1.9 62.0 0.7 12.0 20 1.8 67.4 0.7 12.0 21 1.8 69.4 0.8 13.5 22 2.0 71.4 0.9 14.	2	8.5			
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44 4.1 134.4 2.5 49.6 45 3.8 136.0 2.4 50.5 46 3.9 140.2 2.4 53.2 47 3.9 145.8 2.5 56.6 48 4.1 148.2 2.6 58.2 49 4.0 155.9 2.5 63.0 50 4.0 161.2 2.5 66.4 51 4.1 166.3 2.6 69.6 52 4.1 170.4 2.6 72.2 53 3.7 174.1 2.4 74.6					
45 3.8 136.0 2.4 50.5 46 3.9 140.2 2.4 53.2 47 3.9 145.8 2.5 56.6 48 4.1 148.2 2.6 58.2 49 4.0 155.9 2.5 63.0 50 4.0 161.2 2.5 66.4 51 4.1 166.3 2.6 69.6 52 4.1 170.4 2.6 72.2 53 3.7 174.1 2.4 74.6					
46 3.9 140.2 2.4 53.2 47 3.9 145.8 2.5 56.6 48 4.1 148.2 2.6 58.2 49 4.0 155.9 2.5 63.0 50 4.0 161.2 2.5 66.4 51 4.1 166.3 2.6 69.6 52 4.1 170.4 2.6 72.2 53 3.7 174.1 2.4 74.6					
47 3.9 145.8 2.5 56.6 48 4.1 148.2 2.6 58.2 49 4.0 155.9 2.5 63.0 50 4.0 161.2 2.5 66.4 51 4.1 166.3 2.6 69.6 52 4.1 170.4 2.6 72.2 53 3.7 174.1 2.4 74.6					
49 4.0 155.9 2.5 63.0 50 4.0 161.2 2.5 66.4 51 4.1 166.3 2.6 69.6 52 4.1 170.4 2.6 72.2 53 3.7 174.1 2.4 74.6		3.9	145.8	2.5	
50 4.0 161.2 2.5 66.4 51 4.1 166.3 2.6 69.6 52 4.1 170.4 2.6 72.2 53 3.7 174.1 2.4 74.6					
51 4.1 166.3 2.6 69.6 52 4.1 170.4 2.6 72.2 53 3.7 174.1 2.4 74.6					
52 4.1 170.4 2.6 72.2 53 3.7 174.1 2.4 74.6					
53 3.7 174.1 2.4 74.6					
3.4 1/5.8 2.2 75.7	54	3.4	175.8	2.2	75.7

		DIGESTER=	III-D1	
DAY	RATE (1/D)	TOTAL (1)	CH4 (1/D)	TOTAL CH4 (1)
5 5	4.3	181.1	2.8	79.1
56	4.6	185.6	3.0	82.0
57	4.1	190.3	2.7	85.0
58	3.7	194.0	2.4	87.4
59	2.9	197.2	1.9	89.5
60	2.9	200.0	1.9	91.3
61	4.0	204-2	2.6	94.1
62	4.1	207.3	2.6	96.1
63	4.1	209.5	2.6	97.5
64	4.1	219.8	2.6	104.2
65	4.1	224.2	2.6	107.1
66	3.2	227.6	2. 1	109.3
67	3.0	230-4	1.9	111.1
68	2.9	233.0	1.9	112.8
69	4.5	238.0	2.9	116.0
70	3.9	241.5	2.5	118.3
71	3.1	244.5	2.0	120.2
72	2.5	247.1	1.6	121.9
73	3.0	250.0	1.9	123.8
74	3.3	253.2	2. 1	125.8
75	2.7	256.1	1.7	127.7
76	2.8	259.0	1.8	129.6
77	2.8	260.5	1.8	130.5
78	2.7	262.9	1.7	132.1 134.7
79	2.7	267.1	1.7 1.7	135.8
80	2-6	268.8	1.7	137.5
81	2.6	271.5 274.8	1.5	139.6
82 83	2.3 2.3	277.2	1.4	141.1
84	2.4	279.3	1.5	142.5
85	2.3	281.9	1.5	144.1
86	1.9	283.3	1. 2	145.0
87	2. 1	285-0	1.3	146.1
88	1.9	286.5	1. 2	147.0
89	2. 1	288.6	1.3	148.3
90	2-2	290.6	1.3	149.6
91	2.5	293.0	1.5	151.1
92	2.4	295.4	1.5	152.6
93	2- 1	297.5	1.3	153.9
94	2.0	301.3	1.2	156.2
95	2. 2	303.8	1.4	157.8
96	1.1	304.6	0.7	158.3
97	2.0	305.7	1.3	159.0
98	2.3	309.7	1.4	161.5
99	1.9	311.3	1.2	162.4
100	2.3	313.1	1-4	163.6

		DIGESTER=	III-E1	
DAY	RATE (1/D)	TOTAL (1)	CH4 (1/D)	TOTAL CH4 (1)
1	0.0	0.0	0.0	0.0
2	7.3	2.9	0.3	0.1
3	11.0	12.7	0.6	0.7
4	8.6	21.9	0.7	1_ 4
5 6	7-4	28.4	0.7	2.0
7	•.2 2.7	32.7 33.9	0.5	2.6
8	2.0	37.5	0-4 0-3	2.7
9	2.0	39.3	0.3	3.3 3.6
10	1.4	40.7	0.3	3.9
11	1.0	41.6	0.2	4.1
12	1.5	43.0	0.4	4.4
13	0.8	43.7	0.2	4.6
14	0.8	44.8	0.2	4.9
15	1.0	45.6	0.3	5.1
16	0.8	46.9	0.3	5.6
17	0.5	47.4	0.2	5.7
18	0.6	47.9	0.2	5.9
19	0.6	48.5	0.2	6.1
20	0.5	48.9	0.2	6.3
21	0.5 0.6	49.5	0.2	6.5
23	0.3	50-1 50-4	0.3	6.8
24	0.1	50.5	0.1	6.9 6.9
25	0.3	51.0	0.1 0.2	7.2
26	0.7	51.5	0.4	7.4
27	0.6	51.9	0.3	7.6
28	0.3	52.3	0.2	7.8
29	0.3	52.6	0.2	7.9
30	0-2	52.8	0. 1	8.1
31	0.2	52.9	0_1	8.1
32	0.2	53.1	0. 1	8.2
33	0.5	53.6	0.3	8.5
34	0.2	53.8	0.1	8.7
35 36	0.4	54-1	0-2	8.8
37	0.2	54.3 54.4	0 - 1 0 - 1	8.9 9.0
38	0.4	54.6	0. 2	9.1
39	0.3	54.8	0.2	9.3
40	0.4	55.4	0.3	9.6
41	0.6	55.7	0.3	9.8
42	0-4	56.2	0.3	10-1
43	0.0	56.2	0.0	10.1
44	0.2	56.7	0.1	10_4
45	0.0	56.7	0.0	10.4
46 47	0.0	56.7	0-0	10.4
48	0.3 0.3	57.1	0.2	10.7
49	0.0	57.3 57.3	0.2 0.0	10.8
50	0.0	57.3	0.0	10.8 10.8
51	0.4	57.8	0.0	11.1
52	0.2	58.1	0.1	11. 2
53	0.2	58.2	0. 1	11.3
54	0.0	58.2	0.0	11.3

Appendix B
SUMMARY OF GAS PRODUCTION DATA FROM ALL
DIGESTERS

Digester	Duration	Range of	Total	Methane	Total	
		gas prod.	gas	content	methane	
	(days)	(1/day)	(1)	(%)	(1)	
		•				
I-A	118	0.8 - 5.4	311.9	N/A	N/A	
I - B	46	0.8 - 2.2	46.8	N/A	N/A	
II-Al	70	0.8 - 2.4	97.8	N/A	N/A	
II-A2	7 1	0.8 - 3.3	129.0	N/A	N/A	
II-B1	3 4	0.1 - 0.5	43.0	N/A	N/A	
II-B2	66	0.4 - 1.9	70.4	N/A	N/A	
II-C1	68	0.9 - 1.7	63.6	N/A	N/A	
II-C2	30	0.8 - 1.3	29.2	N/A	N/A	
III-Al	5 4	0.1 - 0.4	51.0	N/A	N/A	
III-A2	54	0.0 - 2.0	48.1	0.0-57.8	7 • 4	
III-A3	5 4	0.1 - 0.6	41.8	N/A	N/A	
III-B1	5 4	0.0 - 0.6	9.5	N/A	N/A	
III-B2	5 4	0.0 - 0.2	2.4	N/A	N/A	
III-B3	5 4	0.0 - 1.7	37.0	14.3-27.2	4.3	
III-C1	5 4	0.0 - 0.2	3.3	N/A	N/A	
III-C2	5 4	0.0 - 0.6	5.7	N/A	N/A	
III-C3	5 4	0.0 - 1.2	42.7	14.5-46.0	7.0	
III-D1	100	1.1 - 4.6	313.1	22.8-65.0	163.6	
III-D2	80	0.3 - 1.4	80.6	N/A	N/A	
III-E1	5 4	0.0 - 1.5	58.2	19.8-62.3		
III-E2	5 4	0.2 - 0.6	60.9	N/A	N/A	
III-E3	54	0.0 - 0.3	25.9	N/A	N/A	
111 23	J 1	0.0	- 3 • 7	,	,	

Note: Range of gas prod. rate (1/day) and methane content (%) are as observed 10 days after starting the digester.

N/A - Data not available

 $\label{eq:Appendix C} \textbf{Chemical Oxygen Demand Of The Residue}$

Digester	COD mg/1
III-A2	24,320
III-B3	17,152
III-C3	17,120
III-D1	8,560
III-E1	15,020