

HIGH SOLIDS ANAEROBIC DIGESTION FOR THE LONG TERM EXPLORATORY
NASA LUNAR SPACE MISSIONS

By

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To the farmers of my country awaiting bioenergy revolution next decade & their service:
my sole inspiration to carry out this study

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TABLE OF CONTENTS

	<u>page</u>
ACKNOWLEDGMENTS.....	4
LIST OF TABLES.....	8
LIST OF FIGURES.....	10
LIST OF ABBREVIATIONS	12
ABSTRACT.....	14
CHAPTER	
1 INTRODUCTION	16
1.1 Background and Justification	16
1.2 Objectives	17
1.3 Thesis Organization	18
2 ANAEROBIC DIGESTION OF SOLID WASTES: A REVIEW	20
2.1 Introduction	20
2.2 Anaerobic Digestion of Biomass	22
2.3 Factors affecting Anaerobic Digestion	24
2.3.1 Temperature	24
2.3.2 pH	26
2.3.3 Pretreatment	27
2.3.4 Digester Designs	28
2.4. Comparison of mixed and unmixed systems.....	31
2.4.1 One Stage Mixed Systems.....	31
2.4.2 One Stage Unmixed systems.....	32
2.4.3 Comparison.....	32
2.4.5 Two Stage Systems.....	33
2.4.6 Hybrid Systems.....	35
2.5 Conclusion	37
3 MATERIALS & METHODS.....	46
3.1 Introduction	46
3.2 Component Samples	46
3.2.1 Human Wastes	46
3.2.2 Packaging	47
3.2.3 Adhered and Uneaten Food	47
3.2.4 MAGS	47
3.2.5 Gray Tape	48

3.2.6	Papers	48
3.2.7	Towels, Washcloths and Fire Retardant Clothing	48
3.2.8	Biodegradable Packaging Materials	48
3.3	Feedstock Preparation.....	48
3.4	Set Up of Biochemical Methane Potential Assays.....	49
3.5	Biogasification System Set Up	51
3.5.1	Anaerobic Digester	51
3.5.2	Biogas Flow Measurement.....	51
3.5.3	Biogas meter operation	52
3.5.4	Calibration of biogas meter	52
3.5.5	Positive Pressure Testing.....	54
3.6	Analysis.....	55
3.6.1	Gas Analysis	55
3.6.2	Liquid Analysis	56
3.6.2.1	pH.....	56
3.6.2.2	Soluble chemical oxygen demand	56
3.6.3	Solids Analysis.....	56
Moisture content	56
Volatile solids	57
4	BIOCHEMICAL METHANE POTENTIAL STUDIES	63
4.1	Introduction	63
4.2	Biochemical Methane Potential of Lunar Wastes	63
4.2.1	Background.....	63
4.2.2	Results and Discussion	64
4.3	Biochemical Methane Potential of Biodegradable Packaging Material	67
4.3.1	Background.....	67
4.3.2	Results	69
4.3.3	Discussion.....	69
4.3.4	Conclusions	72
5	SINGLE STAGE BIOGASIFICATION STUDIES	85
5.1	Introduction	85
5.2	Background.....	85
5.3	Results & Discussion	86
5.4	One Stage Biogasification of Bio Bag	88
5.5	Conclusions.....	89
6	TWO STAGE BIOGASIFICATION STUDIES AND CONCEPTUAL DESIGN	97
6.1	Introduction	97
6.2	Two Stage Biogasification Studies	97
6.2.1	Background.....	97
6.2.2	Results and Discussion	98

6.3	Full Scale Conceptual Design	100
6.3.1	Background.....	100
6.3.2	Reactor Volume Calculations	100
6.3.4	Sizing the second stage of two stage system.....	101
6.3.5	Digester Operations.....	102
6.3.5.1	Thermophilic System.....	102
6.3.5.2	Mesophilic System	103
6.4	Energy Potential of Anaerobic Digestion Operations During 1 Year Exploratory Lunar Space Mission	105
6.5	Energy Requirements.....	106
6.5.1	Energy Required for the Digester Start-Up	106
6.5.2	Heat Losses from Insulation	107
6.5.3	Heat of Vaporization.....	107
6.5.4	Energy Requirement of Pump.....	108
6.6	Comparison of Lunar mission wastes digesters with Mars mission wastes	110
6.7	Conclusion	112
7	CONCLUSIONS AND FUTURE WORK.....	115
7.1	Conclusions.....	115
7.2	Future Work	116
 APPENDIX		
A	BIOGASIFICATION STUDIES FOR NASA: JOHNSON SPACE CENTER- HOUSTON	118
B	PILOT SCALE STUDY: OPERATION OF A SEMI-CONTINUOUS ANAEROBIC DIGESTER UNDER THERMOPHILIC CONDITIONS	121
LIST OF REFERENCES.....		125
BIOGRAPHICAL SKETCH.....		129

LIST OF TABLES

<u>Table</u>	<u>page</u>
2-1 Digester performance for one stage mixed system	38
2-2 Digester performance for one stage Unmixed system.....	41
2-3 Digester performance for multi stage system.....	44
3-2 Formulation of Simulated Synthetic Human Feces.....	58
3-3 Description of biodegradable bags	59
3-4 Composition of stock solutions	59
4-1 Biochemical Methane Potentials of Lunar Wastes.....	74
4-2 Comparison of theoretical and experimental methane yields for Lunar wastes ..	75
4-3 Comparison of theoretical and experimental methane potentials for Lunar wastes.....	76
4-4 Mathis Steam Treatment Results	77
4-5 Biochemical Methane Potentials of Steam Treated Lunar Wastes	78
4-6 Biochemical methane potential of biodegradable bags	79
4-7 Improved methane potential of Lunar wastes with biodegradable packaging	80
5-1 Single Stage Biogasification of Lunar Wastes	91
5-2 Biogasification of Bio-Bag.....	91
6-1 Energy Consumption for Lunar Digesters	109
6-2 Net Energy Gain for Lunar Digesters	109
6-3 Initial water requirement for Lunar Digesters	110
6-4 Estimates of daily solid waste stream for Mars mission (source: Haley et al., 2002).....	111
6-5 Two stage biogasification of Lunar wastes	113
A-1 NASA JSC Landscape waste compositon	118
A-2 NASA JSC Office waste compositon.....	119

A-3	NASA JSC Cafeteria waste compositon	120
A-4	NASA JSC Cumulative Methane Yield Results	120
B-1	Feed analysis of Citrus waste	124

LIST OF FIGURES

<u>Figure</u>	<u>page</u>
1-1 NASA Lunar colony pictures (Source: http://www.nasa.gov/)	19
2-1 Simplified process steps for anaerobic digestion	23
2-2 Types anaerobic digestion processes	29
2-3 Anaerobic digestion process	30
2-4 The scheme of the laboratory-scale one stage mixed anaerobic biogas digester. Q=Quality of a measured value; M=motor; T=Temperature; R=recorded values; I=instrument C=controller. (Source: Demirel et al.,2009)	31
2-5 One stage unmixed system demonstrated by Charles for MSW (Source: Charles et al., 2009)	32
2-6 Two stage biogasification of Indian MSW (Source: Vietez et al., 1999)	34
2-7 SEBAC system (Source: Chynoweth et al., 1993)	36
3-1 Canisters used for steam pretreatment studies	60
3-2 Digester setup for biogasification studies	60
3-3 Biogas U-tube meter	61
3-4 Soda Lime Scrubber	61
3-5 Mathis Labomat used for steam treatment studies	62
4-1 Biochemical Methane Potentials of Individual Lunar Waste Components	81
4-2 Biochemical Methane Potentials of Steam Treated Lunar Waste Components ..	81
4-3 Biochemical Methane Potentials of Biodegradable Bags	82
4-4 Steam treated Lunar waste components exposed for A)2 hours B) 1 hour C)30 min D) 15 min	83
4-5 Degraded biodegradable bags A)Bio Bag B)Bag-to-Nature C)Eco Film D)Eco Safe.....	84
5-1 Comparison of Mixed and Unmixed System for Lunar Waste Biogasification	92
5-2 COD/pH variations in Mixed and Unmixed System	92

5-3	Methane Yield/COD variations in Mixed and Unmixed System	93
5-4	Cumulative Methane Yield for Biogasification of Bio Bag.....	93
5-5	COD/pH Variations in the Biogasification of Bio Bag.....	94
5-6	Methane Yield/COD Variations in the Biogasification of Bio Bag.....	94
5-7	Degraded samples from single stage biogasification of Lunar wastes : A) Packaging material B) Clothes C) Wipes D) Grey Tape.....	95
5-8	Degraded Bio Bag from single stage biogasification	96
6-1	Thermophilic Digester Operation.....	102
6-2	NASA Lunar Thermophilic Digesters: 3 dimensional view	103
6-3	NASA Lunar Thermophilic Digesters: Top view	103
6-4	Mesophilic Digester Operation.....	104
6-5	NASA Lunar Mesophilic Digesters: 3 dimensional view	105
6-6	NASA Lunar Mesophilic Digesters: Top view.....	105
6-7	Space Mission Waste Composition (source: Haley et al., 2002).....	110
6-8	Cumulative Methane Potential of Two Stage System.....	114
6-9	Variation of cumulative methane yield and SCOD in digester for two stage system (Run-1)	114
B-1	Digester set up for pilot scale studies.....	123

LIST OF ABBREVIATIONS

AFR	Anaerobic filter reactor
ALS	Advance life support system
ARS	Air revitalization system
ATCS	Active thermal control system
BVAD	Baseline values and assumptions document
BMP	Biochemical methane potential
BPS	Biogas production system
CH ₄	Chemical formula for methane
CM	Crew Member
COD	Chemical oxygen demand
CSTR	Continuous stirred tank reactor
ECLSS	Environmental control and life support system
ESCSTC	Environmental systems commercial space technology center
ESM	Equivalent systems mass
EVA	Extra vehicular activity
FPS	Food production system
GC	Gas chromatograph
GMO	Genetically Modified Organisms
HAS	Human accommodation system
HRT	Hydraulic retention time
HSLAD	High solids leachbed anaerobic digestion
IFAS	Institute of food and agricultural sciences
ISS	International space station
IVA	Internal vehicular activity

LSS	Life support system
MAGS	Maximum Absorption Garments
MSW	Municipal solid waste
NASA	National Aeronautical and Space Administration
OFMSW	Organic fraction of municipal solid waste
OLR	Organic Loading Rate
OLF	Organic Loading Factor
RT	Retention Time
STP	Standard Temperature and Pressure
SBR	Sequencing batch reactor
SCOD	Soluble chemical oxygen demand
SEBAC	Sequential batch anaerobic composting
SS	Suspended solids
STR	Stirred tank reactor
TS	Total solids
UASB	Up-flow anaerobic sludge blanket
VFA	Volatile fatty acids
VS	Volatile solids
VSS	Volatile suspended solids

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Major: Agricultural and Biological Engineering

"What would it be like to live on the moon?" The National Aeronautical and Space Administration (NASA) solidified its goal of a 'Colonization of the Moon' as a reality in the near future. The Lunar outpost will be an inhabited facility on the surface of the Moon which NASA currently proposes to construct over five years between 2019 and 2024. In a confined environment away from Earth's surface, regeneration of resources including air, water, and nutrients is essential for the crew to survive. NASA is currently funding research for a variety of solid waste resource recovery technologies that may provide useful alternatives to the current method of waste management. One of the biological technologies being tested is a type of anaerobic digestion named high-solids anaerobic digestion.

The technical feasibility of applying high-solids anaerobic digestion for reduction and stabilization of the organic fraction of solid wastes generated during Lunar space missions was investigated. Anaerobic biochemical methane potential assays run on several individual waste feedstocks expected to be produced during Lunar space missions resulted in ultimate methane yields ranging from 0.01 to 0.846L g⁻¹ solids

added. Two batch systems were tested, one of which had no agitation of solids during digestion of materials and the other was mixed continuously at 180 RPM. The unmixed digester performed better than the agitated digester. The anaerobic biodegradation performance of the NASA Lunar mission waste stream was characterized in a two stage hybrid system under thermophilic conditions. Based on these characteristics, a prototype digester was designed and sized for a space mission with a four-person crew during a one year exploratory Lunar space mission.

With a view to increasing methane yield and decreasing undigested solids, two options were investigated: heat treatment of waste and replacement of current plastic packaging material with biodegradable polymers. Steam heat treatment did not show any significant effect in terms of methane potential or degradation. Four different brands of compostable/biodegradable polymers available in the market were tested for their biodegradability and biochemical methane potential. The Bio Bag® showed the most promise. Incorporating biodegradable materials could potentially enhance methane yield by 24% and reduce solid residues by 70%.

Research presented here supports the use of high-solids anaerobic digestion for bioregenerative reduction and stabilization of organic components of solid wastes during extended Lunar space missions.

CHAPTER 1 INTRODUCTION

1.1 Background and Justification

Since the beginning of time, a human has been fascinated by the moon. The National Aeronautical and Space Administration (NASA) solidified its goal of a 'Colonization of the Moon' as a reality in the near future. The Lunar outpost will be an inhabited facility on the surface of the Moon which NASA currently proposes to construct over the five years between 2019 and 2024. Waste treatment and removal for missions to moon will be more challenging due to the longer mission duration regardless of complications from the environment. Waste management for such missions may employ more efficient versions of technologies than developed for Shuttle or completely different approaches may be more cost effective. Depending on the mission protocols, indefinite stable storage for the end products of any waste processing scheme will be necessary.

Historically wastes generated during human spaceflight are materials with no further utility requiring only storage until missions end. However, Exploration Waste Subsystems may reclaim resources from input wastes allowing greater closure within the overall life support system. The waste subsystem collects waste materials from life support subsystems and interfaces. Current NASA spacecraft waste handling approaches essentially rely on dumping and/or storage. For future long duration Lunar mission, it is practically impossible to get all the stored wastes back to the earth and the waste generated over a year cannot be dumped in Lunar surface. The present studies highlights the importance of a technology called 'Anaerobic Digestion' which not only reduces the wastes on the Lunar surface, but may provide significant fuel out of it during

a year of exploration. Anaerobic Digestion (AD) or biogasification is a biological process in which microorganisms break down organic matter into methane and carbon dioxide under anaerobic (or no oxygen) conditions. The technology is ideally suited for space mission, as it does not require oxygen.

1.2 Objectives

The goal of this research was to effectively carry out bench-scale studies on the anaerobic digestion (also known biogasification) of NASA long term Lunar mission waste stream in an effort to identify critical factors and performance measures during batch operation. The research findings would ultimately lead to a proposal of a system design and operation concept for full-scale application of biogasification. This goal was chosen as a sub- study on an on-going project (Biogasification Studies for Johnson Space Center, NASA: High Solids Technology) carried out over twelve months in the Bioprocess Engineering Research Laboratory, Agricultural and Biological Engineering Department, University of Florida, Gainesville. The goals of this research work were divided into four objectives.

- **Objective 1:** Determine the biochemical methane potential of NASA Lunar waste stream and the effect of pretreatment.
- **Objective 2:** Determine the biochemical methane potentials of biodegradable materials as an alternative for Lunar waste packaging.
- **Objective 3:** Evaluate appropriate process designs for anaerobically digesting Lunar mission wastes
- **Objective 4:** Propose a full scale design for anaerobic digestion of Lunar mission waste and carry out mass and energy balances for this system.

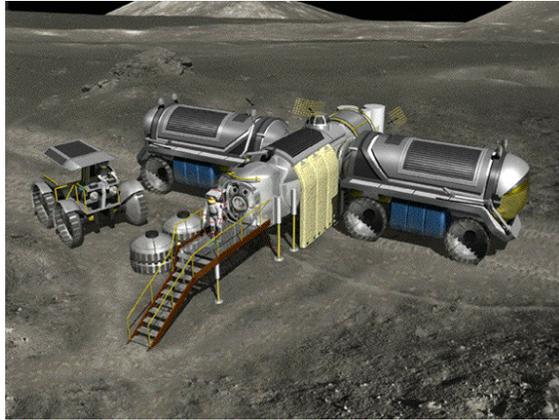
1.3 Thesis Organization

This thesis is divided into seven Chapters. Following this Chapter, the Chapter 2, reviews anaerobic digester designs used for high solids feedstocks like solid wastes and biomass. The purpose of this review was to identify process designs that would be most applicable for Lunar wastes which were then tested in laboratory scale apparatus. Chapter 3, Materials and Methods, includes description of the materials and methods employed to meet the objectives. It lists the assumptions made during the entire analysis and describes the procedures followed during the implementation.

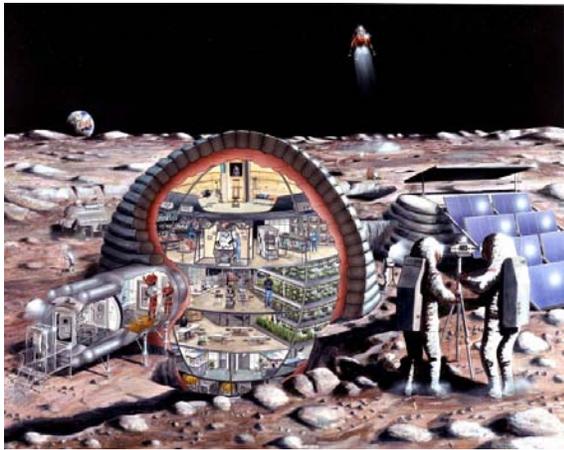
Chapters 4, 5 and 6 describe and discuss the results of the experiments that determined the biochemical methane potential assays as well as the performance of process designs tested for biogasification of Lunar wastes. Chapter 6 also proposes a full scale design for Lunar mission and presents mass and energy balance for this design. Conclusions and Future work, is the Chapter 7 and discusses the future work that is possible in this area.



A



B



C



D

Figure 1-1. NASA Lunar colony pictures (Source: <http://www.nasa.gov/>)

CHAPTER 2 ANAEROBIC DIGESTION OF SOLID WASTES: A REVIEW

2.1 Introduction

The recent oil crisis, global warming concerns and the consequent price rises have spawned considerable interest in the exploration of renewable energy sources. Biomass will be the most significant renewable energy source in the next few decades until solar or wind power production offers an economically attractive large-scale alternative. Biomass may be converted to a variety of energy forms including thermal, steam, electricity, hydrogen, ethanol, methanol, and methane. Selection of the energy form is dependent upon a number of factors, including need for direct heat or steam conversion efficiencies, energy transport, conversion process and hardware, economies of scale, and environmental impact of conversion process waste streams. Under most circumstances methane is an ideal fuel. Currently it represents about 20% of the US energy supply in the form of natural gas (Chynoweth et al., 2001). Related to this, an extensive pipeline distribution system and a variety of hardware are in place for its domestic, municipal, and industrial use. Compared to other fossil fuels, methane produces few atmospheric pollutants and generates less carbon dioxide per unit energy. Because methane is comparatively a clean fuel, the trend is toward its increased use for appliances, vehicles, industrial applications, and power generation. Although some applications require high purity methane, it can be used in a variety of stages of purity and efficiencies of transport and energy from conversion are good. (De Baere et al., 1984). Other fuels such as methanol and hydrogen are not well developed commercially for production and use and are more difficult to produce from biomass. Ethanol is becoming a popular biomass- derived fuel. Although it has the advantage of easy

storage and transport, the fermentation process for its production requires extensive feedstock pretreatment and pure culture maintenance, and energy requirements associated with feed processing and product separation result in overall low process efficiencies. These problems are not characteristic of processes for biological conversion of biomass to methane.

“The literature on anaerobic digestion of solid wastes may at times appear confusing or difficult to summarize, one likely reason is that it is hard to find papers with similar experimental set-ups. In fact, it is precisely the appropriateness of a given reactor design for the treatment of particular organic wastes which forms the focus of most research papers. The comparison of research data and drawing of conclusions is difficult because the great diversity of reactor designs is matched by an as large variability of waste composition and choice of operational parameters (retention time, solids content, mixing, recirculation, inoculation, number of stages, temperature). Empirical knowhow is the rule and there certainly does not exist a consensus over the optimal reactor design to treat solid wastes. The reason most likely lies in the complexity of the biochemical pathways involved and the novelty of the technology” as quoted in Vandevivere et al. (2002). The focus of the present review is to categorize the reactor designs used for solid feedstocks to delineate the effect of mixing, temperature, pH control and retention time etc on the rate of biogasification, extent of degradation and methane yields.

This Chapter surveys the primary biomass sources for methane (CH₄) production reported in the literature. The various operational factors like type of digester, scale of operation, mode of operation, pretreatment, HRT (Hydraulic Retention Time), OLR

(Organic Loading Rate), pH control, methane yield and VS (Volatile Solids) reduction is tabulated for different types of feedstocks. Animal manures, sewage sludges and liquid effluents (<10% solids) from biomass-based industries, which are secondarily derived from the vegetation, are outside the scope of this review. In this review, the extensive literature data have been tabulated and ranked under various categories and the influence of several parameters on the methane potential of the feedstocks are presented. Most of the data reported do not contain any statistical information on variability of methane yield, HRT and OLR etc., only the mean values have been reported. A few of the data from the literature lack homogeneity in conditions of measurement, units, etc. and, in some cases, the data given by individual research groups are inadequate and are not included in this outline.

2.2 Anaerobic Digestion of Biomass

The term biomass would apply to agricultural (or forest) residues, any garbage, refuse, sludge and other discarded material resulting from community activities or commercial operations. Solid waste management has become a major concern in the world recently due to the huge quantities generated world-wide.

Anaerobic digestion is a process in which syntropic consortia of microorganisms break down organic material in the absence of oxygen to produce biogas a mixture of methane and carbon dioxide. Large organic chain molecules such as cellulose and starch are broken down into simpler sugars and monomers. Non-methanogenic populations depolymerize organic polymers and ferment them to acetate, hydrogen, and carbon dioxide. Methanogenic bacteria convert acetic acid, hydrogen, and carbon dioxide to methane (Boone et al., 1993; Smith & Frank, 1988). The digestion process begins with bacterial hydrolysis of the input materials in order to break down insoluble

organic polymers such as carbohydrates and make them available for utilization by microbial consortia. Acidogenic bacteria then convert the sugars and amino acids into carbon dioxide, hydrogen, ammonia, and organic acids. Acetogenic bacteria then convert these resulting organic acids into acetic acid, along with additional ammonia, hydrogen, and carbon dioxide as shown in Figure 2-1. In the absence of methanogens, fermentation products, volatile organic acids and hydrogen build-up. This build-up retards the overall degradative process causing a decrease in pH which inhibits growth and stops fermentation. The overall role of methanogenesis in the biosphere is to complete the degradation process by removal of inhibitory fermentation products (Chynoweth and Pullammanappallil, 1996).

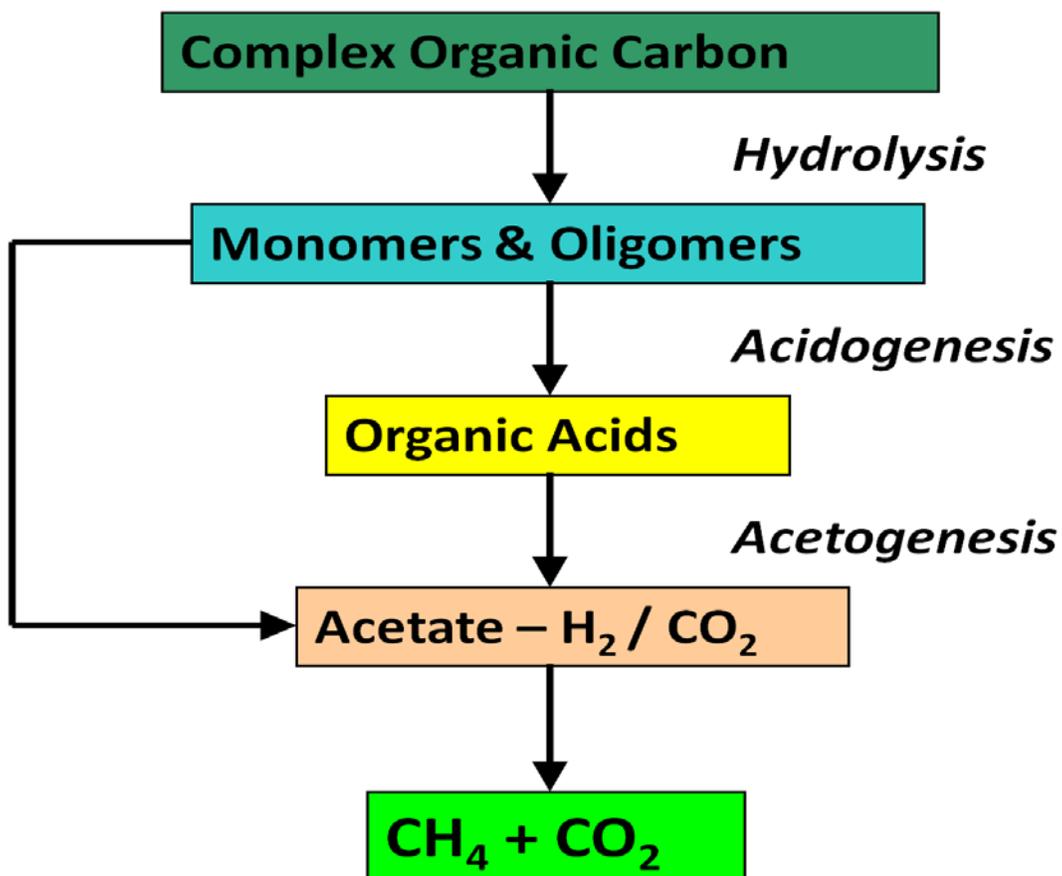


Figure 2-1. Simplified process steps for anaerobic digestion

Anaerobic digestion of solid waste is becoming a popular method to treat these wastes because it can generate biogas as an energy resource. For example, Canada generates approximately 1.45×10^8 t of biomass per year. Anaerobic digestion of these biomass using conventional technologies could generate 1.14×10^{10} m³/year of CH₄ with a heating value of 4.56×10^8 GJ, which is equivalent to about 4.4 % of Canada's current annual energy use (Levin et al., 2007). At the same time, the digested residue from anaerobic digestion could serve as fertilizer for plant growth (Svensson et al., 2004). Moreover, anaerobic digestion has limited impact for our environment (Mata-Alvarez et al., 2000).

2.3 Factors affecting Anaerobic Digestion

The common factors affecting anaerobic digestion process are basically temperature, pH, pretreatment of feedstock and digester design. The performance of the process and the methane yield varies considerably depending on these process factors.

2.3.1 Temperature

Anaerobic digestion may be operated in psychrophilic (12-16 °C), mesophilic (35-37 °C) or thermophilic conditions (55-60 °C). From the present survey, it was found that 88% of the literature preferred mesophilic operations. However thermophilic temperatures, the rates of degradation and biogasification are faster, and have greater potential to destroy weed seeds and plant and human pathogens, which is especially beneficial for reapplying the digested residue with little post treatment back on to the fields to recycle nutrients (Koppar and Pullammanappallil, 2007). For example, in Mace et al. (2003) found that biodegradability of municipal solid waste could be enhanced by thermophilic operation and the corresponding ultimate methane yield was about 10%

higher. The greater energy demand for thermophilic temperature is approximately the same as the excess energy produced in the process in many cases.

All the benefits of thermophilic digestion outlined above are not necessarily applicable under all situations. It appears the feedstock may have an impact on the choice of temperature. Lee et al. (2009) investigated the effect of mesophilic, thermophilic and psychrophilic temperature conditions on anaerobic digestion of kitchen waste. Mesophilic system showed 24% more VS reduction than thermophilic system. While psychrophilic system showed lowest VS degradation. In terms of methane yield, thermophilic system showed the highest followed by mesophilic and psychrophilic. It appears that a greater extent of hydrolysis or solubilization occurred during mesophilic digestion as inferred from the higher VS reduction, but it is likely that not all the hydrolyzed product was converted to methane as inferred from the lower methane yield compared to thermophilic temperature. The nature of inoculum has a bigger impact on the performance of thermophilic digester. Most thermophilic digestion studies (Rich et al., 1995) (Pullammanappallil et al., 200) utilized an inoculum (or starter culture) that was obtained from mesophilic digester. Mesophilic digesters are more prevalent and it is easier to obtain this inoculum. Since dominant species required for thermophilic digestion is not usually found in large numbers in a mesophilic inoculum, this leads to slower kinetics of degradation until the inoculum is well adapted for thermophilic conditions. However, studies in literature do not take into account this adaptation period. For instance, in the studies carried out by Rich et al. (1995), MSW was used as a feedstock in a stirred thermophilic digester operated in batch mode. The process had to be operated for 90 days to obtain a methane yield of 0.398L/g Vs at STP. However,

Rivard et al. (1990), obtained the same methane yield in semicontinuously fed stirred mesophilic digester at a HRT of 20 days. The apparent lack of process stability attributed to thermophilic digestion (Gallert and Winter, 1997) could be due to use of improper inoculum. Therefore, provided thermophilic inoculum is available, the applicability of thermophilic digestion for the feedstock under consideration should be investigated as the benefits of this process temperature range outweigh that at mesophilic temperature

2.3.2 pH

The effect of pH on anaerobic digestion varies between different groups of microorganisms in the digester. pH of 6.8 to 8 is optimum for methanogenesis while a broader pH range from 5 to 8 is optimum for acidogenesis (Anaerobic digestion website, www.anaerobic-digestion.com/, accessed on 10/25/09). It should be noted that pH is not the only cause of inhibition and indeed other substances if present above certain concentrations can inhibit the digestion process (Frostell et al., 1984). Lai (1999) clearly demonstrated that methanogenesis can be initiated fairly quickly in a bed of MSW by providing adequate pH buffer to an extent that prevented significant drops in pH. This was more critical than supplying inoculum to start the digestion process. A deliberately pH inhibited digester was activated quickly by raising the pH above 6.5 (Lai, 1999). It is necessary to maintain pH at an appropriate set point for optimum digestion. Typically the tendency is for the pH to drop below neutral levels due to accumulation of organic acids. To some extent the process itself is able to generate alkalinity/buffer to maintain pH close to neutral levels. This is due to dissolution of carbon dioxide produced in the process, which dissociates to bicarbonate and carbonate ions in turn providing pH buffering capacity. To take advantage of this ability it is necessary to carefully

manipulate the feed and recirculate leachate so as to prevent an excessive accumulation of organic acids or significant drop in pH. Leachate recirculation has been used as a means for pH control. For instance, Chugh et al. (1989) and Charles et al. (2009) demonstrated recirculation of anaerobic digester liquor as a quality pH control measure during anaerobic digestion of MSW. Many commercial technologies (e.g. DRANCO) (Citrus Summary 2004–05) employ leachate recirculation as well as mixing digested residue with feed to provide alkalinity and inoculum during digestion of MSW.

The other option is to maintain pH by dosing chemicals like NaOH (Yoshiyukiueno et al., 2007; Shanmugam et al., 2009; Wilkie et al., 1986), HCl (Gunasselan, 1998), KHCO_3 (Demiral et al., 2009; Rich et al., 1995) and Ca(OH)_2 (Sharma et al., 1988; Saini et al., 1989). Systems employing pH control by dosing chemicals can be operated at greater organic loading rate (OLR). For instance, during anaerobic digestion of MSW, Rich et al. (1995) employed pH control by bicarbonate addition to maintain pH between 7.3-7.4. They were able to achieve OLR of 7.8 g VS/L/d, while studies carried out by Cecchi et al. (1990) on similar feedstock and digester design without any pH control could achieve OLR only up to 2.1 g VS/L/d. Even though pH control by chemical dosing may be advantageous for terrestrial applications, choice of an appropriate design that is able to manipulate pH by controlling feed rate or leachate recirculation may be economical for non-terrestrial applications as this approach does not require hauling chemicals to the lunar base.

2.3.3 Pretreatment

Necessary physical pretreatment steps may include magnetic separation, comminution in a rotating drum or shredder, screening, pulping, gravity separation etc. Ensiling reported to be an advantageous pretreatment for sugar beets (Svensson et al.,

2005). Alkali pretreatment is the most common type of pretreatment for biomass digestion to maintain the optimal pH in the digester (Dar et al., 1987) Thermal hydrolysis or steam hydrolysis is a most common type of pretreatment for synthetic feedstock (Vargas et al., 2009) which has been also used in carrying out the studies on Lunar waste stream in later chapters.

Most of the MSW feedstock were shredded before putting into the digesters. Rich et al. (1995) found that shredding to 0.8mm size is optimal for pilot scale batch stirred tank anaerobic digestion of MSW. Rivard et al. (1990) found out the effect of yeast extraction as a pretreatment on anaerobic digestion of MSW.

In the present studies, pretreatment/preprocessing did not seem to have much more effect on methane yield. For instance, in the anaerobic digestion of MSW, Rich et al.(1995) used the shredded samples for the anaerobic digestion got the methane yield of 0.398 L/g VS which is at par of what Cecchi et al.(1990) got for MSW without any pretreatment and preprocessing. Pretreatment/preprocessing seemed to do well in terms of OLR. Preprocessed MSW samples achieved 70% OLR than the unprocessed in the studies mentioned above. Pretreatment/preprocessing is the unnecessary investment of capital as it has no significant effect on methane yield.

2.3.4 Digester Designs

The anaerobic digestion processes used for biomass feedstock can be broadly classified into three categories: one stage systems, multistage systems and hybrid systems. The biomethanization of organic wastes is accomplished by a series of biochemical transformations, which can be roughly separated into a first step where

hydrolysis, acidification and liquefaction take place and a second step where acetate, hydrogen and carbon dioxide are transformed into methane. In one-stage system, all these reactions take place simultaneously in a single reactor, whereas in two- or multi-stage systems, the reactions take place sequentially in at least two reactors.

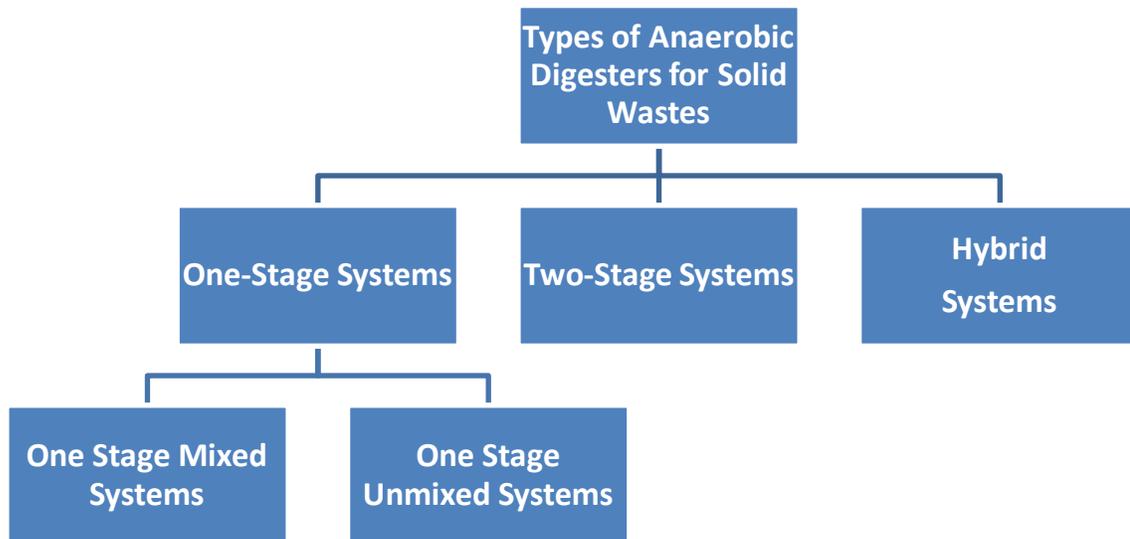


Figure 2-2. Types anaerobic digestion processes

One stage systems can be operated in dry and wet mode. The system with feed having 10-15% TS are termed as 'Wet' systems. The required TS is generally attained by dilution with water. For the systems having more than 15%TS in feed are categorized as 'Dry' systems. Generally MSW are digested in dry systems.

Anaerobic digestion occurs primarily in two steps as shown in Figure 2-3: acid formation and methane formation.

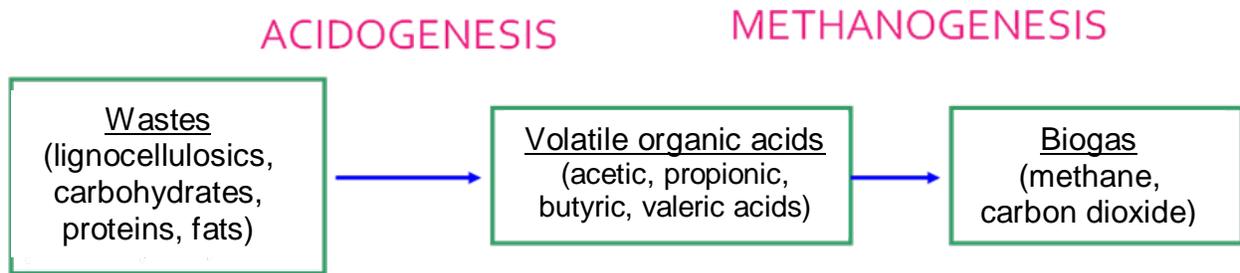


Figure 2-3. Anaerobic digestion process

These processes are mediated by different groups of microorganisms, which require different nutritional compounds and environmental conditions. This could lead to some problems of stability and control if the whole process occurs in one reactor (Demirel and Yenigun, 2002; Pohland and Ghosh, 1971). Therefore, at present more researchers put their efforts into a two phase anaerobic digestion process, which means a physical separation of acid-formers and methane-formers in two separate reactors. In this case, optimum environmental conditions for each group of microorganisms could be provided separately to improve the whole process (Demirel and Yenigun, 2002). Ghosh et al. (2000) showed that given the same operating conditions, the two-phase anaerobic digestion of municipal solid wastes exhibited 18% higher methane yield, 22% higher methane production rate and 13% higher methane concentration than the corresponding performance parameters for one-stage operation. However, others (Weiland et al., 1990) believed that it was unnecessary to treat all kinds of solid wastes in two separate reactors; it depends on the physical and chemical properties of biodegradable wastes. They recommended that one-stage operation could be used to treat solid waste with low protein content such as beet pulp (Weiland et al., 1990).

2.4. Comparison of mixed and unmixed systems

2.4.1 One Stage Mixed Systems

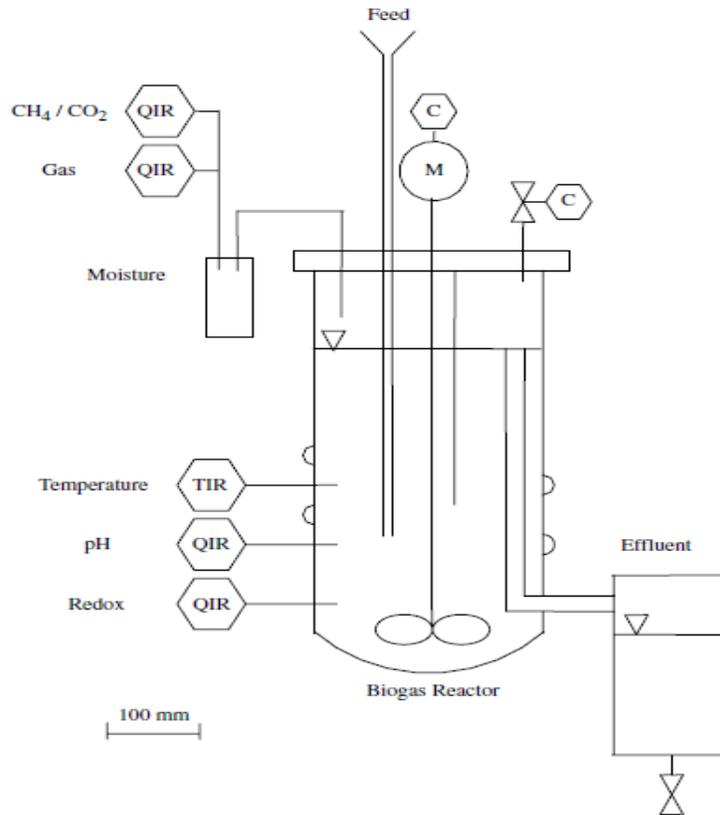


Figure 2-4. The scheme of the laboratory-scale one stage mixed anaerobic biogas digester. Q=Quality of a measured value; M=motor; T=Temperature; R=recorded values; I=instrument C=controller. (Source: Demirel et al., 2009)

One stage mixed systems are the one in which the solids are stirred continuously. Stirring is usually done using agitators, mechanical stirrers, magnetic stirrers etc. The systems in which whole mass of solids are stirred will be termed as mixed systems. If the solids are stationary inside the reactor and only the leachate is being stirred, such systems may not be classified as mixed systems. The mixed system employed by Demirel et al. (2009) for the biogasification of sugar beet silage is shown above.

2.4.2 One Stage Unmixed systems

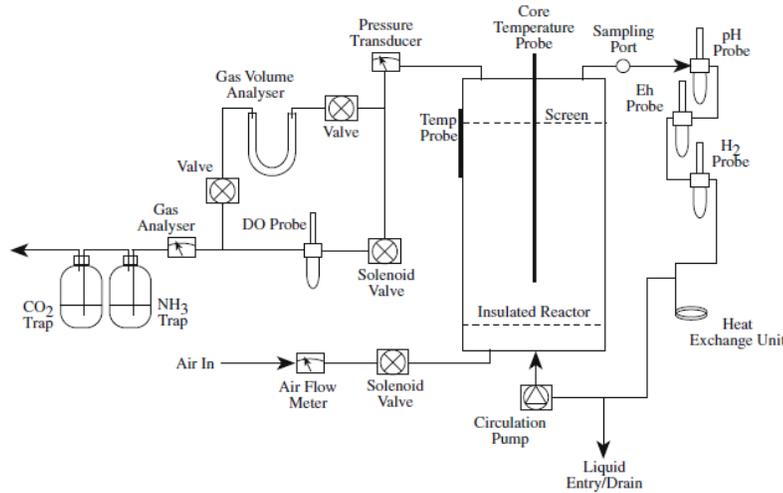


Figure 2-5. One stage unmixed system demonstrated by Charles for MSW (Source: Charles et al., 2009)

One stage unmixed systems are the one in which solids are not being stirred in during the course of digestion. Unmixed systems have advantage over mixed one in terms of capital investment and energy requirement. The wet one stage unmixed system used by Charles for anaerobic digestion of MSW is shown above.

2.4.3 Comparison

The important outcomes are: unmixed system seems to do better than mixed system. For instance, biogasification studies carried out on MSW showed 81.25% more methane yield in unmixed system than mixed system under same biogasification conditions. In terms of OLR, mixed system seemed to do better than unmixed one. For instance, one stage mixed system studies carried out by Cecchi et al. (1990) on MSW achieved OLR of 2.1 g VS/L/d while the same studies carried out by Stenstorm et al. under unmixed conditions achieved OLR of 1.04 g VS/L/d.

2.4.5 Two Stage Systems

The rationale of two- and multi-stage systems is that the overall conversion process of OFMSW to biogas is mediated by a sequence of biochemical reactions which do not necessarily share the same optimal environmental conditions (Vendevivere et al., 2002). Optimizing these reactions separately in different stages or reactors may lead to a larger overall reaction rate and biogas yield (Ghosh et al., 1999). Typically, two stages are used where the first one harbors the liquefaction-acidification reactions, with a rate limited by the hydrolysis of cellulose, and the second one harbours the acetogenesis and methanogenesis, with a rate limited by the slow microbial growth rate (Liu and Ghosh, 1997; Palmowski and Miiller, 1999). With these two steps occurring in distinct reactors, it becomes possible to increase the rate of methanogenesis by designing the second reactor with a biomass retention scheme or other means (Weiland, 1992; Kiibler and Wild, 1992). In parallel, it is possible to increase the rate of hydrolysis in the first stage by using microaerophilic conditions or other means (Capela et al., 1999; Wellinger et al., 1999). The application of these principles has led to a great variety of two-stage designs. The increased technical complexity of two-stage relative to single-stage systems has not, however, always been translated in the expected higher rates and yields (Weiland, 1992). In fact, the main advantage of two-stage systems is not a putative higher reaction rate, but rather a greater biological reliability for wastes which cause unstable performance in one-stage systems. It should be noted however that, in the context of industrial applications, even for the challenging treatment of highly degradable biowastes, preference is given to technically-simpler one-stage plants. Biological reliability is then achieved by adequate buffering and mixing of incoming wastes, by precisely controlled feeding rate and, if

possible, by resorting to co-digestion with other types of wastes (Weiland, 2000). Industrial applications have up to now displayed little acceptance for two-stage systems as these represent only ca. 10 % of the current treatment capacity (De Baere, 1999).

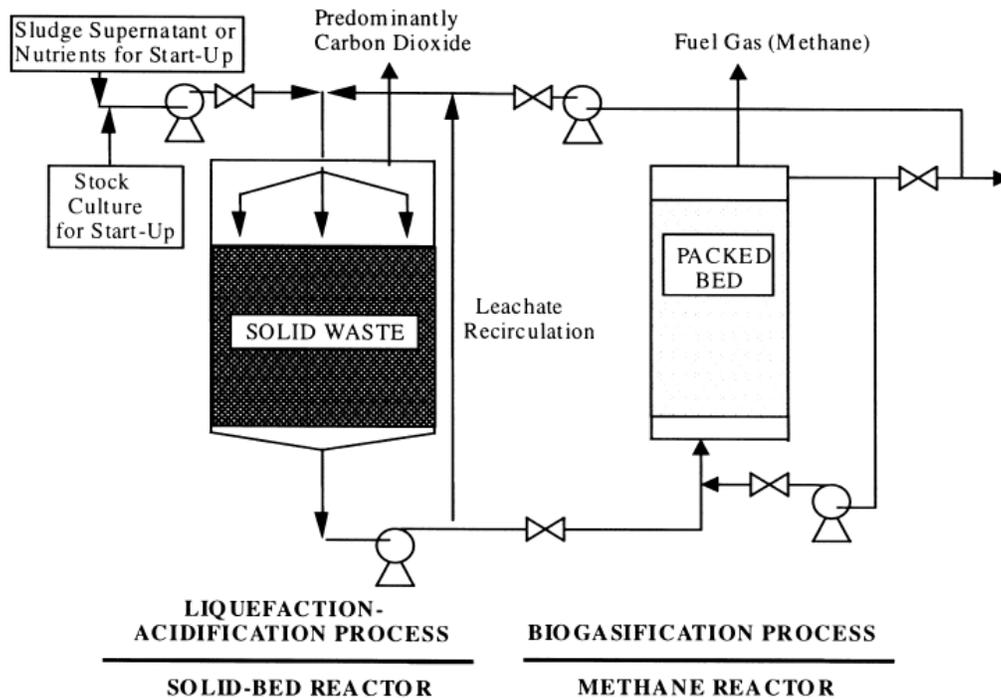


Figure 2-6. Two stage biogasification of Indian MSW (Source: Vietez et al., 1999)

In the two stage biogasification studies carried out on simulated Indian MSW by Vietez et al. (1999), solid bed reactor was used packed with a density of 160 kg/m³. The reactor was charged with 1.227 kg (dry) waste. The waste was chopped into the 2cm size pieces. Fermentation reaction stopped after about 2.5 months of solid bed fermentation at which time total volatile fatty acids concentration accumulate. It demonstrated the methane yield of 0.27 L/g VS after HRT of 295 days and 30% reduction in VS.

In another kind of two stage biogasification studies carried out by Chug et al 1999, two solid leachbed digesters were used. Unsorted MSW shredded to 10cm was fed in

the batch mode at pilot scale in 200 L digester demonstrated the methane yield of 0.18 L/gVS at STP with 54.7% reduction in VS. In a two phase mesophilic biogasification studies carried out on fruit and vegetable wastes by Bouallagui et al. 2004, the anaerobic sequencing batch reactor (ASBR) was used. The reactor was fed semi continuously with shredded feed. It achieved 0.337 L/g VS of methane yield at STP. The HRT varied between acidification and methane reactor by 7 days.

2.4.6 Hybrid Systems

Hybrid systems are multistage system. All the reactors in the hybrid system act methanogenic reactor. Hydrolysis, acidification and liquefaction as well as methane formation takes place in all the digesters of the system. SEBAC is an example of hybrid system.

The SEBAC system is an anaerobic sequential batch digestion process designed to overcome inoculation, mixing and instability problems common of anaerobic reactor designs. A liquid recycle method is used to provide water, nutrients and bacteria to the fresh feedstock. Fermentation products such as volatile acids formed during start-up are removed via the liquid handling system to a mature reactor where they are converted to methane. In doing so, the instability in the start-up reactor is eliminated, as is the need for mixing feed and effluent. Organic matter is decomposed primarily to methane, carbon dioxide, and compost over a residence time of 10-30 days.

The SEBAC system requires a minimum of 3 bioreactors linked through a leachate handling, piping and pumping system. As illustrated in Figure, the anaerobic digestion process used in the SEBAC design involves three stages of digestion that occur sequentially as conversion proceeds. The feedstock is not removed, but passes through different stages over time in the same reactor vessel. In stage 1 of anaerobic digestion,

after the shredded waste is placed into a new stage reactor, leachate will be circulated, providing inoculum, moisture, nutrients and bacteria from the nearly completed mature reactor to the new reactor. The circulation of leachate also removes volatile organic acids (VOA) formed in the new reactor during start-up and conveys them to the mature reactor for conversion to methane and carbon dioxide (biogas). In stage 2, the activated stage, the reactor is methanogenic, and is maintained by recycling leachate upon itself. In stage 3, the mature stage, the reactor acts as a mature reactor and its leachate is recycled with a new reactor for startup.

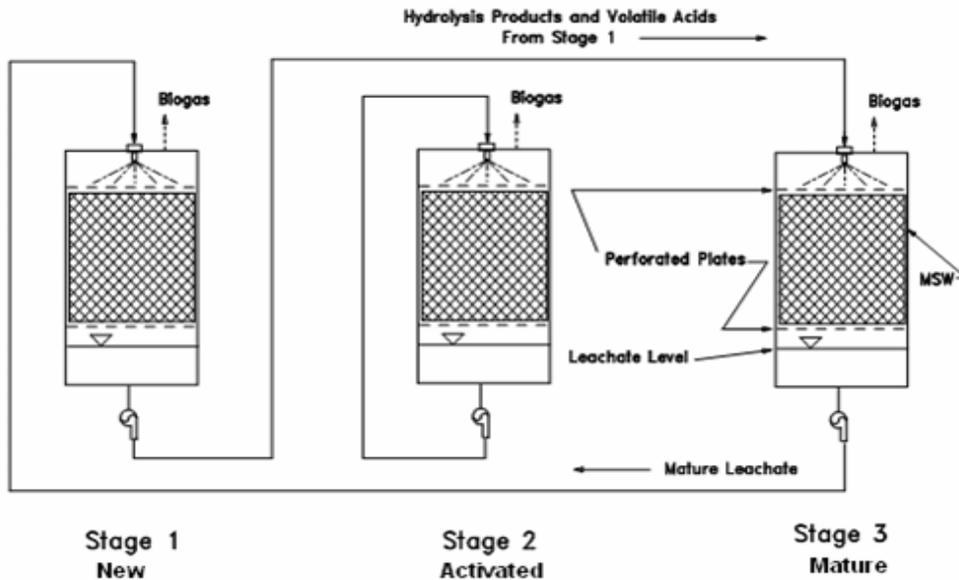


Figure 2-7. SEBAC system (Source: Chynoweth et al., 1993)

The SEBAC process has the advantages of simple operation, low energy requirements and working conditions of low temperature and pressure, while producing methane, carbon dioxide, nutrients, and compost as valuable products. This design of SEBAC was originally intended for terrestrial operation with high solids feeds, such as municipal solid waste. For that application, gravity was relied upon to bring cascading liquid leachate in contact with the organic feedstock by pumping leachate into the top of

the reactor and allowing it to flow by gravity and collect at the bottom for subsequent recycling. In addition, bulk density of solid wastes in the leachbed was kept low to assure sufficient permeability and enhance the leachate percolation rates.

2.5 Conclusion

An extensive literature data on High Solids Anaerobic Digestion has been tabulated and the studies here form the basis for the high solids anaerobic digestion for NASA Lunar wastes. Though most of the literature on anaerobic digestion highlights the use of mesophilic system, the studies on Lunar wastes are carried out at thermophilic conditions as the acclimatized thermophilic inoculum in our lab showed better process performance and stable operations for sugar beet biogasification. The literature review highlights the importance of pH control in improving OLR, but in terms of space operations for future Lunar missions, it will be difficult to carry out chemicals to the Lunar base for pH control. So, Lunar wastes were biogasified with no pH control. Pretreatment did not seem to have much effect in terms of methane potential and it will be difficult in terms of transportation and more capital investment to carry out pretreatment/preprocessing device to the Lunar base. So, no pretreatment or preprocessing was used for anaerobic digestion of Lunar wastes. Lunar wastes were also tested for the one stage mixed and unmixed conditions as well as two stage hybrid operations. Two stage hybrid operations were chosen as it can handle any fluctuations in pH and there was need for pH control during those operations.

Table 2-1. Digester performance for one stage mixed system

Type of Waste	Digester	Scale	Mode	Temp ("C)	Pre-treatment	HRT (days)	OLR kg VS/m ³ /d	pH	pH Control	CH ₄ yield (m ³ /kg VS)	Biogasificatio n Efficiency %	VS red %	Ref
MSW	CSTR	P	B	55	Shredding	90	7.8	7.3 - 7.4	NaHCO ₃ Addition	0.398	91.97	NR	<i>Rich et al.,1995</i>
Sugar beet silage	CSTR	L	C	41-42	Dilution (tap water)	25	7.41	NR	Daily Addition of 1 M KHCO ₃	NR	NR	NR	<i>Demiral e.al.,2009</i>
Sunflower Oil Cake	Erlenmeyer Flasks (250ml)	L	B	35	None	NR	NR	7.1 - 7.6	None	NR	NR	NR	<i>Raposo et al.,2009</i>
OFMSW	CSTR (300 L)	P	S	35	None	25	2.1	NR	None	0.399	98.24	69	<i>Cecchi et al.,1990</i>
MSW	CSTR (3.5 L)	L	S	37	Yeast extraction	20	NR	7.9	KOH addition	0.324	79.26	NR	<i>Rivard et al.1990</i>
Bermuda Grass	CSTR (7 L)	L	S	35	Grinding (<0.5mm)	12	1.6	NR	None	0.219	53.92	37.5	<i>Ghosh et al,1985</i>
Fruit & Vegetable Waste	CSTR (5 L)	L	S	35	Drying (60 C) & Grinding (2mm)	20	2.0	7.8	None	0.400	98.48	NR	<i>Gunaseela n ,2004</i>

Table 2-1. Continued

Jatropha Curcus	CSTR (5 L)	L	S	35	Drying (60 C) & Grinding (2mm)	20	2.0	7.8	None	0.350	86.17	NR	<i>Gunaseelan, 2004</i>
OFMSW	CSTR (300 l)	P	B	35	None	14	4.3	6.9 - 7.3	None	NR	NR	NR	<i>Cecchi et al., 1998</i>
Woody biomass	CSTR (5 L)	L	B	35	Size reduction(0.8 mm)	20	1.6	6.8 - 7.1	None	0.39	96.02	NR	<i>Turick et al., 1991</i>
Fruit & Vegetable Waste	CSTR (10 L) with solid recycling	L	C	35- 37	Hammer milling	NR	3.87	6.2 - 8.0	NaHCO 3 Addition	0.335	82.48	93.2	<i>Lane, 1984</i>
Fruit & Vegetable Waste	CSTR (60 L)	L	S	28- 30	Sun drying & Grinding	20	40	NR	Adjustin g OLR	0.600	150.17	NR	<i>Viswanath et al., 1992</i>
Jerusalem Artichoke	CSTR (10 L)	P	S	37	Ensiling	46	2.6	NR	None	0.307	75.10	61.00	<i>Gunnarson et al., 1985</i>
Gliricidia Leaves	Aspirator Bottles (magnetic stirring) (3 L)	L	B	29- 35	None	NR	NR	6.2	None	0.181	44.56	37.50	<i>Gunaseelan, 1998</i>

Table 2-1. Continued

Napier Grass	CSTR (4 L)	L	S	35	Drying	20	1.23	7.0	NaOH Addition	0.113	27.82	NR	<i>Wilkie et al., 1986</i>
Beet Pulp	STR	P	S	55	Milling	27	5.7	NR	None	0.358	82.00	81.00	<i>Frostell et al., 1984</i>
Water Hyacinth	Upflow STR (5 L)	L	S	25	Size reduction	15	1.6	NR	None	0.420	106.88	NR	<i>Chynoweth et al 1982</i>
Poultry slaughterhouse waste	Stirred acrylic digester	L	S	31	None	50	2.1	NR	None	0.550	137.19	64.00	<i>Salminen et al.</i>
Vine shoots	CSTR (2 L)	L	S	55	NaCl treatment	20	1.0	7.2	None	0.315	72.83	NR	<i>Jimenez et al., 1990</i>

Note:

Table 2-2. Digester performance for one stage Unmixed system

Type of Waste	Digester	S c a l e	M o d e	Te mp. ("C)	Pretreatment	HRT days	OLR kg VS/m 3/d	pH	pH Control	CH4 yield (m3 /kg VS)	Biogasifi cation Efficienc y %	VS red %	Ref
Sugar beet tops & wheat straw	Solid phase reactor	L	B	35	Ensiling	62	NR	NR	None	0.259	63.76	NR	<i>Svensson et al.,2005</i>
	Floating lid reactor (259 m3)	P	B	35	Ensiling	40	1.06 8	NR	None	0.381	93.81	NR	
Korean food waste	Serum Bottle (500 ml)	L	B	35	Crush/screw press	28	NR	7.8	None	0.403	99.22	NR	<i>Lee et al.,2009</i>
Solid municipal sludge	NR	P	B	36	Thermal hydrolysis (170 C)	15	NR	7.8	None	0.580	142.34	53.50	<i>Jolis,2009</i>
	NR	P	B	55	Thermal hydrolysis (170 C)	5	NR	7.5	None	0.860	198.83	61.60	
Swine waste	ASBR (5 L)	L	S	25	Dilution (tap water)	98	2.20	7.5- 7.9	None	0.310	78.89	NR	<i>Garcia et al.,2009</i>
Leather fleshing with MSW	Duran bottles	L	B	35	Minces & homogenized with a commercial blender	35	NR	6.5	addition of either 6 N NaOH or 1 N HCl.	0.457	112.52	68.60	<i>Shanmugam et al.,2009</i>
MSW	Di COM reactor (7 L)	L	B	55	Pre-aeration (48 hrs)	70	7.00	6.5	recirculation of leachate/anaero bic liquor	NR	NR	41.00	<i>Charles et al.,2009</i>

Table 2-2. Continued

Mixture of fresh sugar beet leaves & e ley crop	Water jacketed Plexiglas column reactor	L	S	33	Ensiling	NR	2.00	NR	None	0.360	89.22	NR	<i>Svensson et al., 2007</i>
Beet tops	Stratified bed	P	S	35	Crushing & Ensiling	NR	2.05	NR	None	0.330	81.25	NR	<i>Svensson et al., 2007</i>
Beet silage	NR	L	C	35	Dilution (tap water)	24.8	2.6	7.12	None	NR	NR	NR	<i>Demirel,2009</i>
Tomato processing wastes	Mini B (5 L)	L	S	35	Air drying & powdering	24	4.3	7.0	None	0.420	103.41	NR	<i>Sarada et al. 1994</i>
Silk worm pupae waste	Bioreactor (1.5 L)	L	B	32	Defattation	30	1.0	7.95	None	0.380	95.10	51.00	<i>Viswanath et al.,1994</i>
Straw	Serum Bottle (120 ml)	L	B	35	Ball milling, dilution (tap water)	NR	NR	7.6-7.9	None	0.033	8.13	NR	<i>Hashimoto,1989</i>
Terrestrial weed	Aspirator Bottle (2 L)	L	B	28	Homogenization in blender (0.5 mm sieve size) pretreatment with HCl or NaOH	NR	NR	7.8-8.0	None	0.236	59.46	65.90	<i>Gunaseelan, 1995</i>
Sorghum Cultivars	BMP assay	L	B	35	Grinding (0.8 mm)	NR	NR	7.3-7.5	None	0.400	98.48	92.00	<i>Chynoweth et al.,1987</i>
Lantana Camera	Winchester bottles (3 L)	L	B	31	Alkali pretreatment	NR	NR	NR	None	0.241	60.12	NR	<i>Dar et al.,1987</i>
Mirabilis leaves	Erlenmeyer conical flasks (1 L)	L	B	36	Drying	56	NR	5.4-6.8	None	0.242	59.39	42.60	<i>Sharma et al.,1987</i>

Table 2-2. Continued

Lignocellulosic materials	BMP assay (260 ml)	L	B	35	Shredding	NR	NR	NR	None	0.333	81.99	NR	<i>Tong et al., 1990</i>
Agricultural & Forest residues	Aspirator bottle (5 L)	L	B	37	Grinding	NR	NR	6.7-7.2	using calcium hydroxide (Glaxo).	0.249	60.91	38.7	<i>Sharma et al., 1988</i>
Woody biomass	Aspirator bottle (5 L)	L	B	37	None	NR	NR	6.5-7.1	using Ca(OH) ₂ slurry	0.426	104.21	59.10	<i>Sainai et al., 1989</i>
Calotropis procera leaves	Glass vials (0.1 L)	L	B	35	Shredding & grinding	NR	NR	7.1-8.1	None	0.280	68.94	64.50	<i>Mahamat, 1989</i>
Spent sugar beet pulp	Non stirred reactor	L	B	55	None	7	4.0	NR	None	0.336	77.00	96.00	<i>Koppar et al., 2008</i>
MSW	Solid bed digester (50 G)	P	B	35	Magnetic separation	30	1.04	NR	None	0.560	137.88	NR	<i>Stenstrom et al.</i>
Sugar beet press pulp	Solid Bed Digester (24000 m ³)	F	B	37	Pulping	NR	9.5	NR	None	0.072	20.00	75.00	<i>Brooks et al., 2008</i>
Sorghum, corn, cellulose mixture	Digester (20 L)	L	B	55	None	16.7	10.0	NR	None	0.380	87.86	90.70	<i>Richards et al., 1992</i>
Office paper	Serum bottles (160 ml)	B	B	35	Alkali pretreatment	200	NR	NR	None	0.300	83.33	80.00	<i>Clarkson et al.</i>
Dairy manure	Non stirred tanks	L	C	36	None	2	6	NR	None	0.150	50.00	NR	<i>Jewell et al.</i>
Parthenium solids	NR	L	B	24.2	Drying, homogenization	20	2.06	8.1	Addition of 0.8 N HCl	0.173	44.14	62.42	<i>Gunaseelan, 1998</i>
Cattle manure slurry	Bioreactor (10 L)	L	B	35	Size reduction	10	NR	NR	None	NR	NR	NR	<i>Ong et al., 2000</i>

Table 2-3. Digester performance for multi stage system

Type of Waste	Digester	S c a l e	M o d e	Temp ("C)	Pretreatment	HRT (days)	OLR (kg VS/m ³ /d)	pH	pH Control	CH ₄ yield (m ³ /kg VS)	Biogasificat ion Efficiency %	VS red %	Ref
Unsorted MSW	Solid leachbed (200 L)	P	B	35	Stredding (10 cm)	18	NR	7.0	Leachate recirculation	0.180	50.00	54.70	<i>Chug et al., 1999</i>
Unscreened dairy manure	Non stirred tanks	L	C	36	None	NR	6.0	NR	None	0.151	50.00	NR	<i>Demirer et al.,2005</i>
Solid poultry slaughterhouse waste	Stirred acrylic digester	L	S	31	None	100	2.1	NR	None	0.550	137.20	64.00	<i>Salminen et al.,2002</i>
Mixed unsorted MSW	Solid leachbed (42 L)	P	B	38	Shredding	21	NR	NR	None	0.180	50.00	NR	<i>Clarke et al.,1999</i>
Agricultural residues	Solid bed UASB (Stage 1: 7.6 L) (Stage 2: 2.6 L)	P	B	35	Size reduction	36	4.1	NR	None	0.204	56.67	NR	<i>Parawira et al.,2008</i>
Fruit & Vegetable waste	ASBR	L	S	35	Shredding	3 & 10	1.27	NR	None	0.337	93.61	95.00	<i>Boullagui et al.,2004</i>
Grass	Solid bed upflow	P	B	25	None	190	0.11	NR	None	0.145	40.27	67.00	<i>Yu et al.</i>

Table 2-3. Continued

Potato waste	Solid bed for stage 1 & UASB for stage 2	L	B	37	Size reduction to small pieces in kitchen blender	50	1.26	NR	None	0.390	95.22	95.00	<i>Parawira et al.,2005</i>
White cabbage leaves	Digester (3 L)	L	B	35	None	NR	NR	NR	None	0.382	94.05	NR	<i>Zubr,1986</i>
Fruit & vegetable waste	Up flow sludge bed reactor	L	C	35	None	NR	NR	NR	None	0.383	94.30	90.00	<i>Viturtia et al.,1989</i>
Garbage and paper wastes	CSTR (200 L)for stage 1 & IRPR (500 L) for stage 2	P	C	60 & 55	Pulverization & shredding	8	15.7	5.8-6.0	6.25N NaOH Addition	NR	NR	87.80	<i>Yoshiyukiueno et al.,2007</i>
Simulated Indian MSW	Solid bed reactor	L	B	25 & 35	Chopping into 2cm size pieces	295	NR	NR	None	0.270	75.00	30.00	<i>Vietez et al.,1999</i>
Kitchen garbage	CSTR	L	C	70 & 35	None	4	1.99	7.84	Leachate recirculation	0.306	75.34	61.3	<i>Lee et al., 2009</i>
				55		4	1.01	8.03		0.351	81.15	49.6	
				65		4	1.01	8.00		0.293	65.74	31.8	

Note: L=Lab Scale, P=Pilot Scale=Full Scale, B=Batch, C=Continuous, S=Semi continuous, Temp=Temperature, Red.=Reduction, Ref=Reference

CHAPTER 3 MATERIALS & METHODS

3.1 Introduction

In this Chapter, the materials used to simulate the solid waste stream for long term NASA lunar space missions are described. Two types of experiments were carried out: the biochemical methane potential assay and biogasification studies. The setup for both type of studies are explained along with their operation. The Chapter proceeds in describing the details of the procedure used to carry out various analyses during present studies. The biochemical methane potential studies were carried out in a small serum bottles while biogasification studies were carried out in a 5 liter bioreactor. To see the effect of heat on the biodegradability and methane potential of the samples, steam treatment experiments were carried out. For the steam treatment Experiment, the Mathis equipment shown in Figure 3-5 was used. The Chapter concludes by describing the analytical techniques used to carry out measurements on critical biogasification parameters.

3.2 Component Samples

Based on the Historical Values from previous Shuttle and ISS mission, predicted useful waste products that could be generated from a Lunar mission were simulated in our laboratory. The components of this waste stream is as described in Table 3-1. The description of each component is as follows:

3.2.1 Human Wastes

Formulation of synthetic human waste was adopted from Simulated Human Feces for Testing Human Waste Processing Technologies in Space Systems, Kanapathipillai Wignarajah and Eric Litwiller, Enterprise Advisory Services Inc., NASA-Ames Research

Center (2006-01-2180) as shown in Table 3.2. The goals were to mimic the true water-retention properties of feces and to best fit the chemical composition and consistency reported in literature. By critically evaluating previously used formulations and the composition, both physical and chemical, the stimulant was prepared as reported in 'Simulated Human Feces for Testing Human Waste Processing Technologies in Space Systems' (Wignarajah et al., 2006). The starting chemicals in the synthesis were:

- Cellulose $C_nH_{2n-2}O_n$
- Polyethylene glycol $H(OCH_2CH_2)_n OH$
- Peanut oil $CH - COOH$
- Psyllium powder - Dietary fiber – $C_nH_{2n-2}O_n$
- Miso (Soya powder product) -38% proteins; 21% Fats; 20% fiber; 4% minerals

3.2.2 Packaging

Historical data from previous missions suggests that packaging includes 50% polyethylene and 50% polystyrene materials (Exploration Life Support Baseline Values And Assumptions Document, JSC-64367, DRART July2, 2008). Polyethylene was simulated using an empty milk can while polystyrene was simulated with commercial styrofoam cups .

3.2.3 Adhered and Uneaten Food

Adhered and Uneaten food was quantified to have 27.5% Glucose, 22.5% Fat and 50% Protein. Glucose was simulated with dextrose, fat with squalene and protein with L-isoleucine ((Exploration Life Support Baseline Values And Assumptions Document, JSC-64367, DRART July2, 2008).

3.2.4 MAGS

MAGS are Maximum Absorption Garments to catch metabolic wastes. MAGS were simulated with the wet wipes sent by NASA (catalog No.USAW1070NSDS)

3.2.5 Gray Tape

Gray tapes are made up of 80% polyethylene polymer and 20% butadiene polymer. Scotch electrical gray tapes available in the local stores were used to simulate this.

3.2.6 Papers

Composition of the paper includes cellulose (glucose polymer), wood fiber(with 65.8% glucose, 19.8% xylose, 12.5% galactose and 1.3% mannose). A4 size printing office papers were used to simulate this.

3.2.7 Towels, Washcloths and Fire Retardant Clothing

Towels were simulated with the cotton. Clothes were simulated with the Sear's Fire Retardant Welding Cloth having 95% cellulose with polybenzimidazole as fire retardant.

3.2.8 Biodegradable Packaging Materials

There are various types of biodegradable plastic materials available in the market. A few of these materials for example, poly lactic acid based packaging materials have already been tested in our laboratory for its anaerobic biodegradability (Moreira, 2009). Therefore, other types of biodegradable materials were tested in this study. The materials were tested were in the form of compostable garbage bags as these were easy to obtain. The biodegradable plastic bags samples used for the present studies are those recommended by Biodegradable Products Institute. The description of the products is listed in Table 3-3.

3.3 Feedstock Preparation

Each of the chemicals used for the simulation of lunar waste stream were stored at normal room temperature away from direct exposure to sunlight. MAGS were stored in

the zip lock bags to avoid evaporation of the moisture. Each of the biodegradable garbage bag sample received from the provider was stored at normal room temperature. Samples were shredded into 1mm x 1mm pieces before transferring into the assay bottles for biochemical methane potential studies. No pretreatment or shredding was done for both single stage and two-stage biogasification studies.

For the steam pretreatment experiments, steel canisters as shown in the Figure 3-1 were used. Unshredded sample was fed into each canister with 150 ml of distilled water. The steam treatment at 160°C was carried out in the Mathis equipment shown in Figure 3-5. Canisters were removed from the Mathis at the time intervals of 15 min, 30 min, 1 hr and 2 hrs. Canisters were then allowed to cool to room temperature, all the gases were vented off in the fume hood and the samples were filtered using Whatman filter paper. The samples were air dried for 24 hours and then transferred into the assay bottles.

3.4 Set Up of Biochemical Methane Potential Assays

The Biochemical Methane Potential procedures employed were developed from the anaerobic Warburg test combined with serum-bottle techniques by Owen et al. (1979). Modifications to these procedures are also outlined in ASTM (1992) and examples of results can be found in Owens and Chynoweth (1993). The BMP assay was conducted with Corning No.1460, 500 ml serum bottle. Each bottle was fed with 5 g (total weight) of the shredded samples.

In each serum bottle, 200 ml of inoculated media (inoculum and nutrient solution) was added to the 5 g of sample. Bottles were sealed with rubber serum caps of appropriate size. Sealed bottles were inverted and incubated at 55°C. Each assay was accompanied with blank controls containing only inoculated medium. Each component

of the Lunar waste mix was tested. Shredded samples of feedstocks were anaerobically incubated, in a sealed serum bottle, with the standard media and inoculum until gas production had ceased.

Each solution used to make the anaerobic media possess a specific function for the overall success of creating an ideal anaerobic environment. Stock solution (S-1) containing resazurin, a redox indicator, assures the media is in the reduced state and turns the media pink when oxygen is present (Chynoweth and Owens, 2000). Stock solution (S-2) contains macronutrients that assure nitrogen, phosphorus and potassium are not limiting. Stock solution (S-3) contains micronutrients that assure appropriate trace metals are available in the final media. Previously, the micronutrient solution lacked a source for nickel (Owen et al., 1979) but studies showing the importance of nickel in methanogen metabolism resulted in its addition to the defined media (Chynoweth and Owens, 2000). Sodium sulfide solution, a reducing agent, is included in another stock solution (S-4) and serves to remove any remaining available oxygen in the media after preparation. Sodium bicarbonate, the final chemical added, provides pH buffering to assure acidification of the substrate does not cause an inhibitory pH drop. Concentrated stock solutions were used for preparing the defined media as suggested by Owen et al. (1979) and are stored at 4°C. The defined media contains nutrients and vitamins for mixed anaerobic cultures. The composition is tabulated in Table 3-4.

This process can take up to 30 days for simple substrates, such as sugars and starches, and up to 120 days for recalcitrant lignocellulosic substrates, such as cypress (Chynoweth and Owens, 2000). Single bottles of 'Bio Bag', 'Bag-to-Nature' and 'Eco

Film' while duplicate bottles of 'Eco-Safe' were assayed for biochemical methane potential.

3.5 Biogasification System Set Up

3.5.1 Anaerobic Digester

Two types of digesters were used in these experiments: mixed and unmixed. A digester was constructed by modifying a Pyrex glass jar. The volume of the digester was 5 liters. The digester was sealed with a top lid, using an O-ring fitted for gas and liquid tightness and clamped with a stainless steel clamp. Three ports were provided at the top of the lid, one for gas outlet, and others for sample withdrawal. The digester was also equipped with an outlet at the bottom from which liquid samples were collected. No additional mixing device was applied for the unmixed system. Magnetic stirrer was used for mixed stirred reactor system. The digester was placed in an incubator where the temperature was maintained at 55°C. The digester set-up is shown in Figure 3-2.

3.5.2 Biogas Flow Measurement

Many conventional technologies exist when gas flow measurement is a parameter of interest. Diaphragm, rotary and turbine gas meters are common in many industrial and commercial applications, but are limited for high and steady flow conditions. In these studies, the biogas flow rate is low which is not detected by these flow meters. Raw biogas produced from anaerobic digestion of organic matter can cause erroneous flow readings on conventional devices due to moisture and other impurities in the biogas and flow that is intermittent and delivered in packets. For such a purpose, a special U-tube gas meter was used to efficiently measure the gas flow by liquid displacement. (Figure 3-3).

3.5.3 Biogas meter operation

A liquid displacement flow meter (U-tube design) was used to measure biogas flow from digester. This design circumvents the deficiencies of the conventional meters by having error free operation even if gas flow is intermittent, high in moisture and contains impurities and also for low flow rate. The active components of the circuit include a 3-way solenoid valve, a float switch, an electromechanical counter, a time delay relay and a U-tube monometer component. A low volatility fluid antifreeze brand was filled inside the U-tube and the entire apparatus was sealed properly. The biogas from the reactor accumulated in one limb of the U-tube and displaced the liquid inside; when the liquid in the second leg rose to a certain level, the float switch tripped, causing three events to occur simultaneously: a signal was sent to the counter to record the reading for display; the biogas from the first leg was vented into the atmosphere, causing a reset of both liquid levels in both legs; and a timer kept the vent line open long enough to equilibrate the levels. During the vent cycle, the reactor's biogas was isolated from the gas meter. With each switch closure, the counter continued to increment the amount of gas flowing through the meter; cumulative counts per given period would yield a volumetric gas flow rate.

3.5.4 Calibration of biogas meter

Biogas flow was measured by determining the relationship between the counter increment and the volume of incoming gas required to trigger one counter increment. To simulate biogas, which primarily consists of two gas-phase components (methane and carbon dioxide), a specialty, high purity standard was used; 60.00% CH₄ and 40.00% CO₂. A glass syringe of known volume and accuracy (100 ± 1 mL) was used to determine the amount of simulated biogas required to induce one counter increment.

Calibration protocol included injecting a series of simulated biogas doses into a biogas meter via a sealed septum and observing at what volume switch closures occurred. Protocol was conducted in both off-line mode (stand-alone gas meter) and on-line mode (gas meter connected to reactor vessel) during low or no biogas production. The final result of the calibration was an input-output relationship, called a calibration factor with units- mL of gas/count. The precision of calibration factors were characterized by reporting the standard deviation of a population of repeated measurements. Typically, a series of ten injections were deemed as adequate population for determining a gas calibration factor. Values of 55 ± 3.2 mL per count were obtained regularly during calibration protocols. This level of measured resolution (one gas click) on each gas meter was sufficient to provide insight about biogas production trends within a period of study (7-120 days).

In gas measurement applications, the relationship between intensive properties (e.g., temperature and pressure) and gas behavior were considered. The ideal gas law can be applied to real gases when absolute pressures are lower than an atmosphere and when temperatures are not close to the liquefaction point. With near ambient pressures and a 55°C operating temperature, this Equation of state was adequate in characterizing and predicting the behavior of biogas.

The strong relationship between gas temperature and volume received attention during calibration of biogas meters. During experimentation, biogas was produced at 55°C in each vessel and measured externally at a lower temperature. As a result, a cooling affect translated to a variable delivery of volume of gas than what actually was produced in each vessel. To take account of measurement errors due to gas cooling, a

conservative correction factor was implemented in all measurements: normalizing measured gas to standard temperature and pressure (STP) conditions. This factor was conservative because it assumed that gas was collected at 55 °C. The final calibration factor was multiplied by a correction factor ($= 273.15 \div 328.15 \text{ °C}$) to conservatively estimate gas produced in each biogasification vessel.

3.5.5 Positive Pressure Testing

The performance of biogasification experiments was initially evaluated by the quantity of biogas produced per given time. Biomass is mineralized to a methane and carbon dioxide gas mixture from available substrate (solid feedstock and soluble constituents) and released from the bed by buoyancy; subsequently, measurements of gas mixture volumes and composition provide explicit insight to biogas production rate and implicit insight to biochemical progression, respectively. With performance measure being so highly dependent on gas collection, efforts were taken to correctly seal and minimize gas leaks. Possible leak areas considered were as follows:

- Fittings for gas outlet (at top of lid)
- U-tube meter
- Biogas tubing (vessel-to-meter line)
- Top-lid gasket

The leak test consisted of pressurizing the vessel and gas meter system to comparable values seen during biogasification experiments. Each system was injected with air through the biogas sampling septum and the liquid-level in the biogas meter was monitored. Enough air was injected to enable the displaced liquid column to just fall short of tripping the float switch. The level of the fluid in the in-going column was marked to detect changes over time; liquid soap was applied at the aforementioned leak areas to detect any leaks.

3.6 Analysis

3.6.1 Gas Analysis

Assay bottles were periodically analyzed for gas production and composition for more than 120 days. Gas-volume sampling and removal during incubation was performed with glass syringes (5-30 ml depending on gas volume) equipped with 23-gauge needles. Readings are taken at the room temperature and the syringe is held horizontal for measurement. Volume determinations are made by allowing the syringe plunger to move (gently twirling to provide freedom of movement) and equilibrate between bottle and atmospheric pressure. The gas samples were analyzed with a Model 1200 Fisher Gas Partitioner. The GC was fitted with two 6-foot *Haysep* 80/100 mesh columns containing Porapak Q support. Ultra high purity Helium (99.99%) was used as the carrier gas at an operating head pressure of 15 psi. The gas was analyzed for its methane, carbon dioxide, nitrogen and oxygen content. The GC was calibrated with an external standard containing N₂:CH₄:CO₂ in volume ratio of 25:45:30.

Calculations

After each sampling, the value of the measured volume of methane produced by the bottles was converted to dry gas at 1 atm and 0°C (STP) and added to the previous measurements. This cumulative methane volume removed was added to the methane (dry at STP) present in the headspace of the bottle to determine the total cumulative methane volume of the sampling time. The total cumulative methane volumes were corrected for methane production attributed to the medium and inoculum by subtracting the averaged blank control volume from each bottle's total cumulative methane volume. Finally, the corrected cumulative methane yield was calculated by dividing the corrected volume by the weight of sample added to each bottle.

3.6.2 Liquid Analysis

3.6.2.1 pH

The analysis of pH was conducted using the Campbell Scientific pH probe.

3.6.2.2 Soluble chemical oxygen demand

The soluble chemical oxygen demand (SCOD) analysis was carried out using HACH's United States Environmental Protection Agency (USEPA)-approved dichromate method. The method utilized small micro vials that contained the necessary reagents (silver, chromium and mercury) to carry out the analysis. Mixed culture samples were taken at the beginning and the end of the run; each sample was centrifuged (Fisher Marathon micro H centrifuge), filtered (Whatman micro filter paper, 45 μm) and stored for COD analysis. Vials (HACH COD of range: 2 to 1500 mg/L) were filled with mixed culture sample (diluted if estimated detection limit was approached) and digested for 2 hours at 150°C in a COD reactor (HACH, Model 45600). The SCOD of the digested samples were estimated by measuring its color intensity using a colorimeter (HACH, DR/890) against a blank. Average error of colorimetric COD analysis was quantified as $\pm 4\%$ for samples that range 0 to 20,000 mg/L.

3.6.3 Solids Analysis

Moisture content

The moisture content of each aliquot was determined by placing the sample in a constant temperature oven at $105 \pm 1^\circ\text{C}$ for a period of 24 hours. Subsequently, each sample was allowed to cool down to room temperature and weighed with an analytical balance. The percent total solids and moisture was calculated by mass difference.

Volatile solids

After a sample was dried for moisture content and total solids, the volatile solids content was determined. Each sample was placed in an evaporation tray (aluminum) or crucible and then placed inside a furnace at $550 \pm 5^{\circ}\text{C}$ for two hours. After heat treatment, each sample was removed and allowed to cool down at room temperature in a desiccator, before being weighed. The volatile solids content was calculated by mass difference.

Table 3-1. Quantification of NASA Lunar waste stream

S.N.	Type of Waste	Dry Weight kg/(CM-D)	Water Content kg/(CM-D)
1	Human Wastes	0.123	0.090
2	Packaging	0.220	--
3	Adhered Food	0.098	0.070
4	Uneaten Food	0.249	0.210
5	MAGS	0.173	0.058
6	Gray Tape	0.033	--
7	Paper	0.105	0.08
8	Towels & Washcloths	0.100	0.009

CM = crew member; D = day

Table 3-2. Formulation of Simulated Synthetic Human Feces

S.N.	Component	% weight	Weight in grams
1	Yeast Extract	30	1.4850
2	Cellulose	15	0.7425
3	Polyethylene Glycol	20	0.9900
4	Psyllium Husk	5	0.2475
5	Peanut Oil	20	0.9900
6	Miso	5	0.2475
	Proteins (38%)		
	Fats (21%)		
	Fiber (20%)		
	Minerals (4%)		
7	Inorganics	5	
	KCl (40%)		0.0990
	NaCl (40%)		0.0990
	CaCl ₂ (20%)		0.0495
8	Dried Coarse Vegetable Matter		0.0500

Table 3-3. Description of biodegradable bags

S.N.	Sample	Dimension	Weight
1	Bio Bag	37 in x 13 in	30.5 g
2	Bag to Nature	24 in x 30 in	3.19 g
3	Eco Film	17 in x 16 in	9.6 g
4	Eco Safe	38 in x 15 in	40.3 g

Table 3-4. Composition of stock solutions

Stock Solution	Composition	Concentration(g/L)	Volume	
S1	S1	sample	2	
	S2	Resazurin	1	1.80 mL
S4	S3	(NH ₄) ₂ HPO ₄	26.7	5.40 mL
		CaCl ₂ .2H ₂ O	16.7	27.00 mL
		NH ₄ Cl	26.6	
	S4-1	MgCl ₂ .6H ₂ O	120	
		KCl	86.7	
		MnCl ₂ .4H ₂ O	1.33	
		CoCl ₂ .6H ₂ O	2	
	S4-2	H ₃ BO ₃	0.38	2.70 mL
		CuCl ₂ .2H ₂ O	0.18	
		Na ₂ MoO ₄ .2H ₂ O	0.17	
ZnCl ₂		0.14		
NiCl ₂ . 6H ₂ O		0.05		
S4-3	NaVO ₃ . nH ₂ O	0.05		
	H ₂ WO ₄	0.007	0.27 mL	
S5	S5	FeCl ₂ .4H ₂ O	370	18.00 mL
S6	S6	Na ₂ S.9H ₂ O	500	18.00 mL
		Biotin	0.002	1.80 mL
		Folic Acid	0.002	0.90 mL
		Pyridoxine hydrochloride	0.01	
		Riboflavin	0.005	
		Thiamin	0.005	
		Nicotinic acid	0.005	
		Pantothenic acid	0.005	
		p-aminobezoic acid	0.005	
		Thioctic acid	0.005	
S7	S7-1	B12	0.0001	0.18 mL
		Sodium bicarbonate		8.40 g



Figure 3-1. Canisters used for steam pretreatment studies

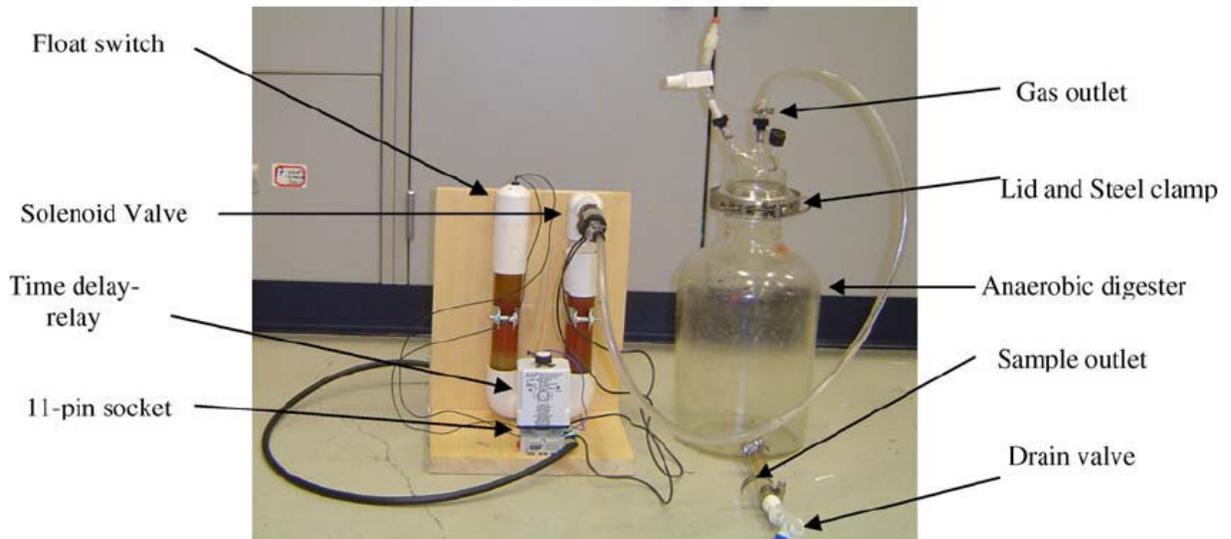


Figure 3-2. Digester setup for biogasification studies

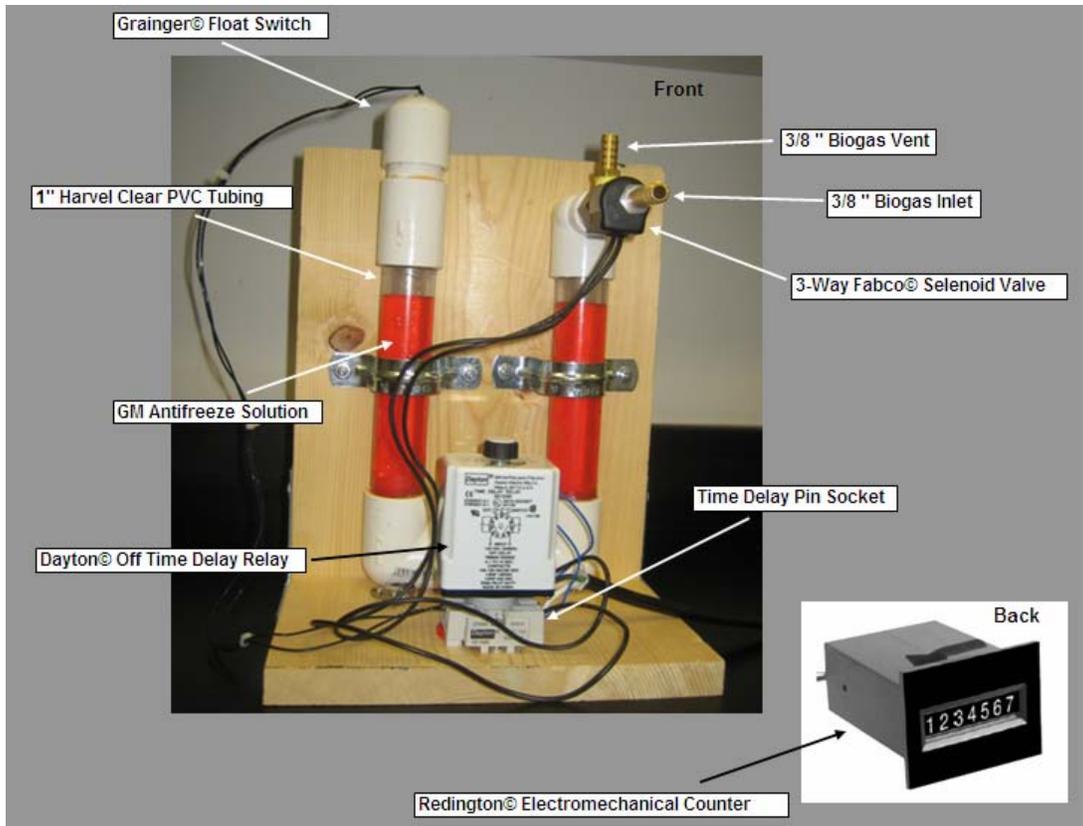


Figure 3-3. Biogas U-tube meter



Figure 3-4. Soda Lime Scrubber



Figure 3-5. Mathis Labomat used for steam treatment studies

CHAPTER 4 BIOCHEMICAL METHANE POTENTIAL STUDIES

4.1 Introduction

The first study of the research work was to determine biochemical methane potential of the individual components in Lunar waste stream at thermophilic conditions. The aim of this study was to analyze the anaerobic biochemical methane assays to determine extent and rate of bioconversion. Further, four different brands of compostable/biodegradable garbage bags available in the market namely Bio Bag, Bag-to-Nature, Eco Film and Eco Safe were tested for their biodegradability and biochemical methane potential under anaerobic condition. . These bags represented biodegradable packaging materials that could potentially replace some of the current plastics used in a Lunar mission.

4.2 Biochemical Methane Potential of Lunar Wastes

4.2.1 Background

Biochemical methane potential is a measure of sample biodegradability under anaerobic digestion conditions. Bioassay techniques are essential for determining biodegradability since no chemical procedure is available which distinguishes between biodegradable and non-biodegradable organics. Bioassay techniques can also measure the presence or absence of inhibitory substances and offer the most promise for resolving anaerobic treatment problems because they are relatively simple and inexpensive and do not require knowledge of specific inhibitory substances.

Both continuous (and semi-continuous) and batch feed techniques have been used to evaluate biodegradability. The continuous procedures closely simulate full-scale anaerobic operation; however they are costly in terms of facilities, equipment, time, and

personnel. Batch bioassay techniques do not have these limitations and thus permit the evaluation of a wide range of variables. Batch techniques can evaluate the influence of shock loads, but, in general do not simulate the effects of real systems as well.

Anaerobic serum bottles containing samples, defined media, and seed inocula are incubated at the desired temperature, and respective gas productions are monitored volumetrically using the syringe method of Nottingham & Hungate (1969). The liquid and gas phases can be sampled periodically by syringe extraction for subsequent analyses.

4.2.2 Results and Discussion

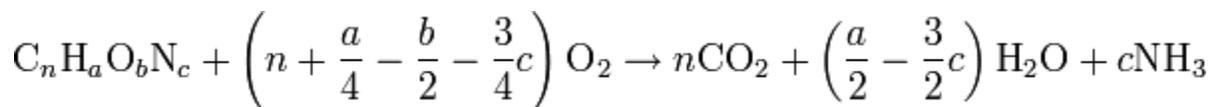
The biochemical methane potential assays were carried out on individual NASA Lunar waste components as described in Chapter 3 with 5 g of each component and 200 ml of mixed culture together with nutrient media. The profiles of cumulative methane yield of individual NASA Lunar waste components are shown in Figure 4-1 and the results tabulated in Table 4-1. Human waste and the food waste seem to be two major components having highest methane potential. The cumulative methane potential of human waste was 0.856 liters of methane per gram dry weight of waste at standard condition of temperature and pressure. Food waste followed the same trend with 0.481 liters of methane per gram dry weight of waste at standard conditions. Packaging materials (which includes mainly polyethylene and polystyrene), wipes and grey tape did not seem to have any significant methane potential. Papers, cotton and Lunar clothing showed methane potentials of 0.237, 0.046 and 0.013 liters of methane per gram of waste at standard conditions, respectively.

It took 54 days for human waste to achieve 95% of its methane yield and 78 days for food waste. Cotton and clothing seemed to degrade faster compared to other Lunar

waste components achieving 95% of their methane potential in 29 days and 20 days respectively. Papers and wipes took significant time to degrade and demonstrated 95% of their methane yield in 66 and 54 days respectively.

The undigested samples from the assay were filtered, dried at ambient temperature for 24 hours and weighed. The degradation was calculated based on the amount of undigested sample recovered compared to what was fed. Human waste and food waste were the only soluble components in the Lunar waste stream and showed 100% degradation after digestion. Papers and cotton showed degradation of 98% and 96% respectively. The rest of the components did not show any significant degradation. Human waste and food waste seemed to degrade all their Soluble Chemical Oxygen Demands (SCOD) at par of what is predicted theoretically and demonstrated their suitability for anaerobic digestion. Papers and clothing, most of which primarily made up of cellulose, also showed good performance in anaerobic digestion process. As tabulated in Table 4-1.

Theoretical COD of all Lunar waste components and the comparison of theoretical and experimental methane yields are reported in Table 4-2. The basis for theoretical COD estimation was that nearly all organic compounds can be fully oxidized to carbon dioxide. The amount of oxygen required to oxidize an organic compound to carbon dioxide, ammonia, and water is given by:



COD was estimated using the Equation:

$$COD = (\text{Moles of } O_2 \text{ required}) * (\text{Molecular Weight of } O_2) / (\text{Molecular Weight of Sample})$$

Human waste has a theoretical methane yield of 0.686 L/g, but experimental methane yield for human waste was 0.856 L/g. The reason for experimental methane yield to be more than theoretical yield may be because of the empirical formula predicted for human waste was not indicative of the actual one. For food waste the theoretical and experimental methane yields were 0.507 and 0.481 L/g respectively, which represents some components in food waste were not completely degraded during the duration of the assay. Packaging material and grey tape seems to have very high methane yield of 1.148 L/g theoretically because of the long polymeric chain they, but experimentally (as expected) there was no significant methane production from these assays. Papers and cotton showed the methane yield at par with their theoretically predicted methane yields. The methane potentials for the Lunar wastes components are tabulated in Table 4-3 and compared with the theoretical predictions. Experimental methane potentials of all the components added up to 315.81 L/CM-D compared to the theoretically predicted 681.88 L/CM-D.

Steam treatment of the Lunar waste components was carried out as described in Chapter 3. Each of 5 g of undigested and a digested sample recovered from single stage anaerobic digestion were steam treated for a period of 15 min, 30 min, 1 hour and 2 hours. The steam treated samples are shown in Figure 4-2. The profiles of the cumulative methane yield of the steam treated Lunar waste components is shown in Figure 4-2. There was 38-40% solubilization for undigested samples after steam treatment. This was not the total mass reduction but the particulate matter solubilization. The SCOD of the undigested samples after steam treatment was very high in the range of 17.4-21.2 g/L and the pH was in the range of 6.06-6.74. One

important observation was that higher the treatment time less is the SCOD and pH. This was probably because the longer exposure to steam might have hydrolyzed the waste. These results were at par of the cumulative methane potential studies in which the sample steam exposed for 15 min showed 86.27% more methane yield than that with 2 hours of steam exposure. The cumulative methane yield for the undigested steam treated samples was in the range of 0.014-0.102 liters of methane per gram of sample at standard conditions of temperature and pressure.

The steam treated digested residues from the digester was tested for its degradability and methane potential in the biochemical methane potential assays. The opposite trend was observed in terms of weight reduction in steam treatment experiments. Solubilization increased with exposure time. Extent of solubilization was in the range of 2-14%. The steam treated samples released less SCOD in the range of 1.12-3.08 g/L which was consistent with extent of solids solubilization. The methane potential of the steam treated digested residues was not more than 0.001 liters of methane per gram of sample at standard condition of temperature and pressure which demonstrates that solid residues coming out of the digester did not have much methane potential and some alternative methods must be developed to degrade it and recover energy out of it.

4.3 Biochemical Methane Potential of Biodegradable Packaging Material

4.3.1 Background

As discussed in the biochemical methane potential studies of individual Lunar waste components, packaging material did not degrade and contribute to methane potential. It is possible that the packaging materials in Lunar waste stream could be replaced with various biodegradable polymeric materials. Other studies in our laboratory

have determined the extent of degradability and methane potential of poly lactic acid materials under anaerobic digestion conditions. To test the biodegradability and methane potentials of other biodegradable polymeric materials, compostable biodegradable garbage bags were used as representative of such materials.

The studies carried out here may be useful for the terrestrial MSW facilities also. Synthetic polymeric plastic materials accumulate in the environment at a rate of 25 million metric tons per annum. Polyethylene (PEs) represent 64% of plastic materials produced as packaging and bottles, which are usually discarded after only brief use. Plastic bags accumulate in the environment due to their low degradability, generating pollution and taking space in landfills. Also, because they have very small masses and are usually contaminated, recycling is economically unfeasible. Their elimination at composting plants is not complete; therefore fragments of bags end up contaminating the compost and ultimately require screening or other processes for their removal. (Scott et al.). Consequently, they are not suitable for recovering energy by incineration, because the heat is lost in evaporating the water instead of producing electricity. The compostable fraction of M.S.W. (Municipal Solid Waste), such as kitchen scraps, grass cuttings, waste from canteens, restaurants, the organic part of solid urban waste (also known as the "wet part") packed in the normal polythene bags is not exposed to the microbial population in the landfill unless the bags are shredded before landfilling. Recently there are a few brands of biodegradable garbage bags available in the market. These biodegradable garbage bags will biodegrade completely and safely leaving no residues and are certified for their biodegradability by ASTM D6400 standards. As the conditions in land filling process are similar to that of anaerobic digestion process, these

biodegradable garbage bags are tested for the degradability and energy potential under anaerobic conditions in the present studies. Now a days, anaerobic digestion is becoming a preferred option for MSW management. Therefore, it is important to investigate the degradability of these compostable materials under anaerobic conditions.

4.3.2 Results

Table 4-4 summarizes the performance of BMP assays in terms of corrected cumulative methane yield (expressed as liters of methane gas produced at STP per grams of the garbage bag sample fed). Duration of the assays varied from 106 to 172 days. Each of assay was fed with 5 g of sample initially. The amount of the sample degraded and the percentage of degradation is reported in Table 4-4. The initial SCOD was 5.0 g/L. The residual SCOD at the end of the run and the final pH is also reported. Table 4-4 also summarizes the duration to produce 95% methane yield potential of each sample as well mass of biogas recovered. Figure 4-3 shows the plot of cumulative methane yield values versus duration of assays for all the samples.

4.3.3 Discussion

The results highlight the agreement between the amounts of biogas produced to the amount of substrate solubilized which validated the mass balance of the process. For instance, 5 g of Bio Bag fed in the process was solubilized completely. Mass of biogas recovered was 5.74 g. About 1.70 g of Bag-to-Nature sample from which 1.90 g of biogas was produced. 1.20 g degraded for Eco Film giving 1.26 g of biogas and 2.30 g of Eco Safe degraded yielding 3.29 g of biogas. It's interesting to note that in all the cases amount of biogas recovered is more than the amount of substrate degraded.

Post run liquid analysis suggests that pH values of all the assays were almost in the range of 7.0 to 8.0 which is the optimal range for methanogenesis. Also, the residual SCOD from all the assays were in the range of 2.48 g/L to 6.54 g/L which highlights that there was no unusually high accumulation of Volatile Fatty Acids (VFAs) indicating stable digestion in all the assays.

Amongst the samples used for the biochemical methane potential analysis, Bio Bag had the most methane potential. The methane potential of Bio Bag is 0.344 L/g while those of the others ranged between 0.084 to 0.146 L/g which is far lower than Bio Bag. Correspondingly extent of solubilization of Bio Bag (95%) was more than that of other samples (23 – 34%). The manufacturer of Bio Bag claims to be the world's largest brand of 100% biodegradable and 100% compostable bags which was validated in the present study.

Next best product in terms of methane potential and degradation was 'Bag-to-Nature' with methane potential of 0.146 g/L and 34% degradation in 127 days. In terms of rate of methane production also Bio Bag seems to do well than others. Duration to achieve 95% methane potential for Bio Bag was 45 days while for others it was observed in the range of 118-165 days. ASTM standards for compostable plastics (ASTM D 6400-04) requires plastic to degrade more than 90% under controlled conditions. Bio Bag seems to meet those standards even under anaerobic digestion conditions. Bio Bag is a blend of GMO (Genetically Modified Organisms) free starch polymer and other renewable resources. Other products like Bag-to-Nature, Eco Film, and Eco Safe even though meets ASTM D 6400 standards for compostability it does not degrade very well under anaerobic digestion conditions. Bag-To-Nature is made

from a blend of organic biopolymers which apparently degrades completely leaving no residues when composted. But, the present studies highlights that 66% of the Bag-to-Nature sample was still undegraded even after 127 days of digestion. Eco Film contains no polyethylene and is heat and moisture stable. Still, Eco Film seems to degrade 24% in thermophilic conditions. Eco Safe claims that its Compostable trash bags are specifically engineered to quickly degrade in 10 to 45 days and fully biodegrade in less than 6 months. It seems to degrade only 23% even after 172 days of digestion.

An interesting trend was observed in the cumulative methane yield profile of Bio Bag, Bag-to-Nature and Eco Safe assays. There is a continual increase in methane production for 10 -20 days following a decrease in methane production for 3-5 days. Again methane production continues to increase for next 10-20 days. While in case of Eco Film assay the methane yield profile increases constantly. The reason for the trend may be due the presence different kind of additives, fillers, copolymers etc used in the production of these bags. Accessibility to the degradable components may require solubilization or degradation of other components. So once a component is degraded it is followed by a lag phase while the other component is exposed for microbial attack. Another reason could be that microbial growth on degradable components may follow diauxic profiles where degradation of one component is initiated after the degradation of another component. Since the exact composition of these bags was not revealed by the manufacturers it was difficult to pinpoint the reasons for the observed profiles. In some cases degradation is activated by heat, UV light and enhanced by mechanical action. (Vargas et al.) showed that PLA pretreatment by irradiation (gamma source and e-beam) or steam (120°C for 3 h) enhanced its degradation under composting

conditions. Likewise, to promote anaerobic degradability it may be necessary to subject these materials to some form of pretreatment.

It should be noted that under current waste management practices where the MSW is mostly landfilled, these “biodegradable” materials may not degrade completely. The present studies also aim to serve high solids anaerobic digestion systems for NASA Long Term Lunar Space Missions. With no oxygen condition on the lunar surface, anaerobic digestion is an option for treating solid wastes. . Bio Bag being the most biodegradable plastic that was tested and with the highest methane potential, can be recommended for the use as packaging material, trash collector as well for coatings on paper and other degradable substrate in long duration space missions. Substitution of packaging materials with Bio Bag type material, the methane potential of a Lunar waste stream can be improved by 24% as demonstrated in Table 4-7.

4.3.4 Conclusions

Individual Lunar waste components were tested for their biodegradability and methane potential. Human waste and food waste demonstrated highest methane potential of 0.856 and 0.481 L/g at STP and complete biodegradability. Paper, cotton and clothing also showed good methane potentials and biodegradability. Other components such as packaging materials, wipes, grey tapes etc did not degrade. Steam treatment did not show any significant effect in terms on methane potential or degradation. Solid residues from the digester did not have much methane potential and some alternative methods must be developed to degrade it and recover additional energy.

Four different brands of biodegradable garbage bags available in the market namely Bio Bag, Bag-to-Nature, Eco Film and Eco Safe were tested for their

biodegradability and biochemical methane potential under anaerobic digestion condition. Bio Bag observed to be best amongst the four in terms of methane potential, biodegradation and rate of methane production. The amount of biogas produced seems to be in correlation with the amount of material biodegraded under balanced digestion condition. Bio Bag achieves 0.344 L/g of methane yield under standard condition of temperature and pressure with 95% degradation in 106 days under anaerobic condition which seem to be in accordance with ASTM D 6400-04 standards for Compostable Plastics. Bio Bag type material when incorporated in long term space missions for packaging could increase the methane potential of lunar waste by 24%.

Table 4-1. Biochemical Methane Potentials of Lunar Wastes

S.N.	Sample	Gas Analysis	Solids Analysis		Liquid Analysis		Duration to produce 95%CH4 yield potential (Days)
		Cumulative CH4 Yield (L/g)@STP	Solubilization/ Degradation (g)	Soulubilization/ Degradation (%)	Residual SCOD (g/L)	Final pH	
1.	Human Waste	0.856	5.0	100%	12.56	8.01	54
2.	Uneaten/Adhered Food	0.481	5.0	100%	12.32	8.41	78
3.	Packaging	0.000	0.1	2%	5.36	7.96	NA
4.	Wet Wipes	0.037	0.3	6%	4.50	8.20	54
5.	Dry Wipes	0.000	0.1	2%	4.20	7.86	NA
6.	Grey Tape	0.004	0.1	2%	3.54	8.02	15
7.	Papers	0.237	4.9	98%	3.12	7.80	66
8.	Cotton	0.231	4.8	96%	7.04	7.69	29
9.	Clothing	0.013	1.7	34%	3.80	7.87	20

Table 4-2. Comparison of theoretical and experimental methane yields for Lunar wastes

S.N.	Sample	Molecular Formula	Molecular Weight (g)	Theoretical COD (g O ₂ /g sample)	Theoretical CH ₄ yield (L/g)	Experimental CH ₄ yield (L/g)
1	Human Waste	C ₄₂ H ₆₉ O ₁₃ N ₅	921	2.049	0.686	0.856
2	Uneaten/Adhered Food	C ₆ H ₁₂ O ₆ C ₅ H ₉ O ₂ C ₆ H ₁₅ O ₂ N ₂	150.57	1.515	0.507	0.481
3	Packaging	C _n H _{2n}	14	3.428	1.148	0.000
4	MAGS	CH ₂ -CH(COONa)	93	0.946	0.317	0.037
5	Gray Tape	C ₂ H ₄ +C ₅ H ₁₀	49	3.428	1.148	0.004
6	Papers	C ₆ H ₁₂ O ₆ C ₅ H ₁₀ O ₅	170.55	1.065	0.357	0.237
7	Cotton	(C ₆ H ₁₀ O ₅) _n	162	1.185	0.397	0.231
8	Clothing	C ₆ H ₁₂ O ₆ C ₁₁ H ₁₅ N ₂	181.15	1.132	0.379	0.013

Table 4-3. Comparison of theoretical and experimental methane potentials for Lunar wastes

S.N.	Sample	Expected Dry Wt (kg/CM-D)	Theoretical CH ₄ Potential (L/CM-D)	Experimental CH ₄ Potential (L/CM-D)
1	Human Waste	0.123	84.38	105.28
2	Uneaten/Adhered Food	0.347	175.93	166.91
3	Packaging	0.220	252.56	0
4	MAGS	0.173	54.84	6.40
5	Gray Tape	0.033	37.88	0.13
6	Papers	0.105	37.49	24.89
7	Cotton & Clothing	0.100	38.80	12.20
		TOTAL	681.88	315.81

Table 4-4. Mathis Steam Treatment Results

S. N.	Sample	Treatment Period (min)	Solids Analysis				Liquid Analysis	
			Original Weight (g)	Wt of the solids remaining (g)	Solubilization (g)	Solubilization % 	pH	COD (g/L)
1	Undigested-1	15	5.0	2.9	2.1	42%	6.74	21.28
2	Undigested-2	30	5.0	3.1	1.9	38%	6.48	19.96
3	Undigested-3	60	5.0	3.0	2.0	40%	6.28	19.32
4	Undigested-4	120	5.0	3.1	1.9	38%	6.06	17.48
5	Digested-1	15	5.0	4.9	0.1	2%	8.05	3.08
6	Digested-2	30	5.0	4.7	0.3	6%	7.43	2.12
7	Digested-3	60	5.0	4.6	0.4	8%	7.15	1.12
8	Digested-4	120	5.0	4.3	0.7	14%	6.43	0.00

Table 4-5. Biochemical Methane Potentials of Steam Treated Lunar Wastes

S.N	Sample	Gas Analysis		Solid Analysis		Liquid Analysis		Duration to produce 95%CH4 yield potential (Days)
		Cumulative CH4 yield @STP (L/g)	Solubilization (g)	Solubilization %	Residual SCOD (g/L)	Final pH		
1	Undigested-1	0.210	3.20	64%	3.80	6.70	45	
2	Undigested-2	0.130	3.05	61%	3.54	6.52	45	
3	Undigested-3	0.065	3.24	64%	3.80	6.43	45	
4	Undigested-4	0.028	3.18	63%	3.12	6.15	45	
5	Digested-1	0.001	1.20	24%	2.12	7.98	25	
6	Digested-2	0.001	1.48	29%	2.08	7.92	25	
7	Digested-3	0.001	1.34	26%	2.24	7.56	25	
8	Digested-4	0.001	1.68	33%	2.80	7.23	25	

Table 4-6. Biochemical methane potential of biodegradable bags

S . N .	Sample	Duration (Days)	Gas Analysis		Solids Analysis		Liquid Analysis		Duration to produce 95% methane yield potential (Days)	Mass of Biogas recovered (g)
			Cumulative CH ₄ Yield (L/g) @STP	Degradation (g)	Degradation (%)	Residual SCOD (g/L)	Final pH			
1	Bio Bag	106	0.344	4.75	95%	5.72	8.09	45	5.74	
2	Bag-to-Nature	127	0.146	1.70	34%	6.54	7.86	118	1.90	
3	Eco Film	127	0.084	1.20	24%	2.48	7.87	120	1.26	
4	Eco Safe	172	0.136	2.30	23%	3.54	8.29	165	3.29	

Table 4-7. Improved methane potential of Lunar wastes with biodegradable packaging

S.N.	Sample	Experimental CH ₄ Potential (L/CM-D)	Improved CH ₄ Potential with Biodegradable Packaging (L/CM-D)
1	Human Waste	105.28	105.28
2	Uneaten/Adhered Food	166.91	166.91
3	Packaging	0	75.68
4	MAGS	6.40	6.40
5	Gray Tape	0.13	0.13
6	Papers	24.89	24.89
7	Cotton & Clothing	12.20	12.20
TOTAL		315.81	391.49

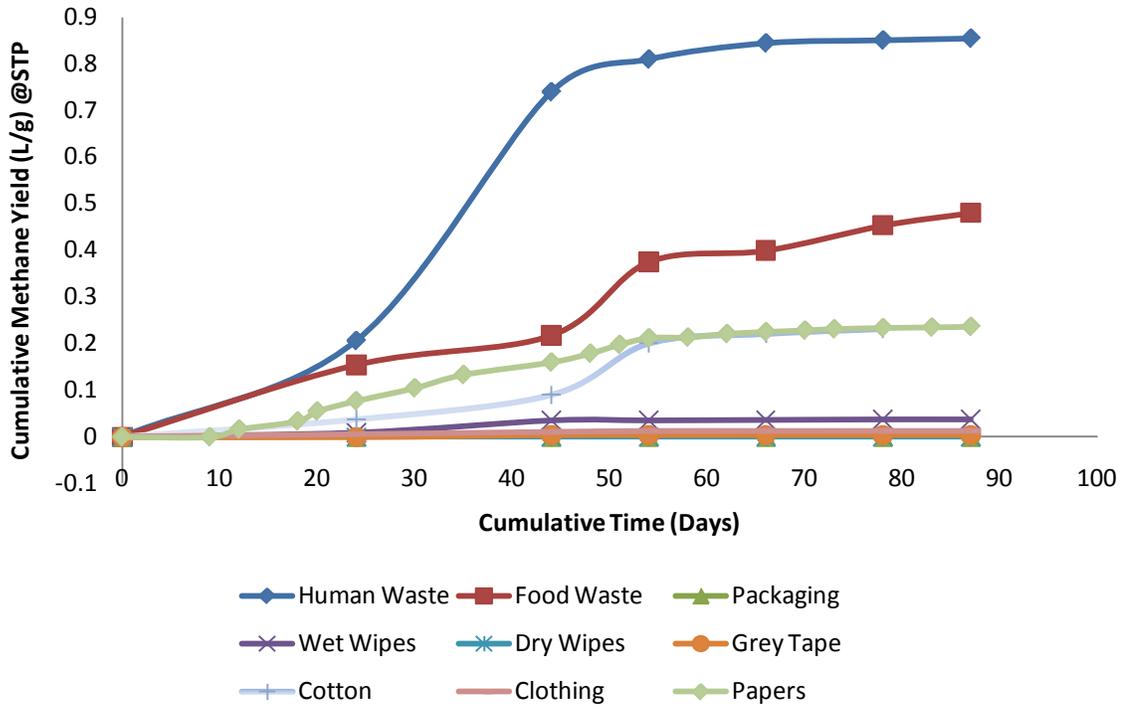


Figure 4-1. Biochemical Methane Potentials of Individual Lunar Waste Components

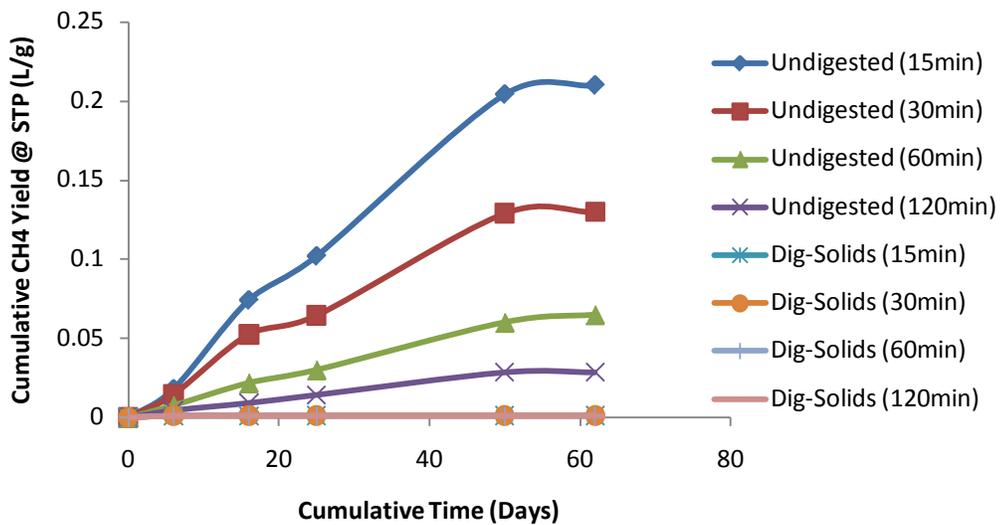


Figure 4-2. Biochemical Methane Potentials of Steam Treated Lunar Waste Components

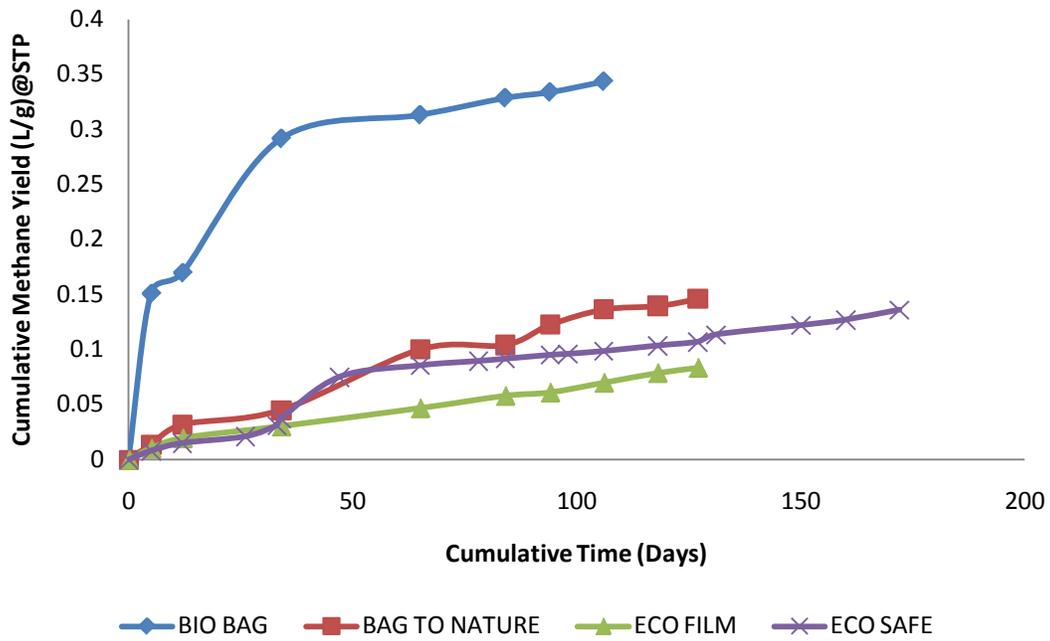


Figure 4-3. Biochemical Methane Potentials of Biodegradable Bags



A



B



C



D

Figure 4-4. Steam treated Lunar waste components exposed for A)2 hours B) 1 hour C)30 min D) 15 min



A



B



C



D

Figure 4-5. Degraded biodegradable bags A)Bio Bag B)Bag-to-Nature C)Eco Film D)Eco Safe

CHAPTER 5 SINGLE STAGE BIOGASIFICATION STUDIES

5.1 Introduction

This part of the study involves batch, single stage, and thermophilic anaerobic digestion of NASA Lunar waste stream. This first iteration of experiments was chosen for its simplistic design and operation. Two batch systems were introduced, one of which had no agitation of solids during digestion materials and the other was mixed continuously at 180 RPM. The progression of an Experiment was measured by the evolution of methane with time. After the feedstock was degraded the reactor was opened and the residue removed and analyzed. The aim of this study was to characterize the anaerobic biodegradation potential of NASA Lunar waste stream as well Biodegradable bags and its methane potential (measured as methane yield)

5.2 Background

Among various technologies that are available for anaerobic digestion, continuously stirred tank reactors (CSTR) are typically used to process high solid materials. Thorough mixing of the content in the digester distributes microorganism uniformly and improves mass and heat transfer, and therefore is regarded as essential in high rate anaerobic digestion. Furthermore, agitation helps particle size reduction and release of produced biogas from the digester contents. Mixing is usually accomplished by mechanical mixers, slurry recirculation or biogas recirculation. The significance of mixing in anaerobic digestion has been reported by many researchers (Kim et al., McMahon et al., Smith et al., Stroot et al.), though contradiction is found in determining the effect of mixing duration and intensity on anaerobic digester performance. Nevertheless, most studies agreed that high intensity mixing had an adverse effect on

digester performance (Kaparaju et al., 2008; McMahon et al., 2001; Stroot et al., 2001; Vavillin et al. 2004.). Batch process offers advantages that mixing process does not have. Batch process is considered energy conserving for it does not require fine shredding of substrates or agitating of digester contents. Anaerobic digestion in batch system can be carried out at ambient pressure and at mesophilic and thermophilic temperatures both. Studies had showed vigorous mixing was harmful to anaerobic digestion (Kaparaju et al., 2008; McMahon et al., 2001; Stroot et al., 2001.) and the negative effect was interpreted as high shear forces disrupting microbial flock structures and disturbing syntrophic relationship between microbes (Vavillin et al. 2004). Therefore, appropriate mixing rate need to be maintained.

5.3 Results & Discussion

The profiles of cumulative methane yield for NASA Lunar waste stream in mixed and unmixed digester is shown in the Figure 5-1. Run1 for both mixed and unmixed system was started with the 100 g dry weight of the Lunar waste combination as mentioned in Chapter 2. Working volume of 4 liters was maintained in both the digesters. The methane production rate and the methane flow rate were monitored for 12 days of digestion. Total methane obtained at standard conditions of temperature and pressure at the end of the run was 1.76 liters for mixed system and 3.46 liters for unmixed system. Cumulative methane yield at the standard conditions of temperature and pressure was 0.035 liters of methane per gram dry weight for mixed system and 0.039 liters of methane per gram dry weight for unmixed system. Daily methane production rate picked at $0.490 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ for mixed digester on day 9 and $1.36 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ for mixed digester on day 6. The daily methane production rate dropped to $0.04 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ for mixed digester and $0.16 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ for unmixed digester when Run 1 was completed.

For mixed digester to achieve its 95% of methane yield potential required 6 days while that for unmixed digester required 5 days.

Run 2 was started with the fresh inoculum for both mixed and unmixed system with the 25 g dry weight of the Lunar waste combination. Working volume of 2 liters was maintained in both the digesters. The methane production rate and the methane flow rate were monitored for 28 days of digestion. Total methane obtained at standard conditions of temperature and pressure at the end of the run was 3.12 liters for mixed system and 4.55 liters for unmixed system. Cumulative methane yield at the standard conditions of temperature and pressure was 0.12 liters of methane per gram dry weight for mixed system and 0.18 liters of methane per gram dry weight for unmixed system. Daily methane production rate peaked at $0.130 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ for mixed digester on day 21 and $1.915 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ for mixed digester on day 2. The daily methane production rate dropped to $0.025 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ on day 10 for mixed digester and $0.024 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ for unmixed digester when Run 2 was completed. For mixed digester to achieve its 95% of methane yield potential required 17 days while that for unmixed digester required 3 days.

In all two runs, unmixed digester exhibited higher methane yield and methane production rate than mixed digester. Only difference was that run1 showed unstable digestion because of overloading compared to run2. In run1, unmixed digester achieved 11.43% more methane yield than mixed digester, while in run 2 unmixed digester achieved 50% more methane yield than mixed digester. In run 1, the degradation of solids obtained in the mixed and the unmixed digesters were 44.80% and 42.00% respectively. While that in the run 2, it was 57.20% and 56.40% respectively. The

results highlights the direct correlation between the amounts of biogas produced to the amount of substrate degraded which validates the mass balance of the process. The validation of mass balance justifies the validation of the biogasification.

The profiles for SCOD for mixed as well as unmixed digesters for run 2 are shown in the Figure 5-3. SCOD concentration in both the digesters initially accumulated, reached the maximum and decreased linearly to a minimum. Mixed digester showed a significantly higher SCOD than unmixed digester. The higher degradation rate of SCOD in unmixed digester is consistent with the higher daily methane production rate. The pH of all the digesters was well in between the range of 7.84 – 9.12 which signified there was no accumulation of any volatile organic acids.

5.4 One Stage Biogasification of Bio Bag

Two experimental runs were conducted in this study. In run1 digester was loaded with 50 g (wet weight) of Bio Bag and maintained at 55 °C during the run. 2 kg of bulking materials ((lava rocks from landscaping supplier, 0.025 m in average size) was added into digester along with the feedstock to prevent compaction and floatation. The digester was inoculated with mixed culture as mentioned in Chapter 3. The first run was ended when the gas production was low enough. The digesters were emptied and washed thoroughly. Residual Bio Bag was discarded, and the digester liquor was kept for the second run. Run 2 was started with the digester liquor from the first run and the digester was loaded with 20 g (wet weight) of Bio Bag sample.

The profiles of cumulative methane yield for the biogasification of Bio Bag is shown in the Figure 5-4. Working volume of 3.5 liters was maintained in both the runs. The methane production rate and the methane flow rate were monitored for 60 days of digestion for run 1 and 30 days for run 2. Total methane obtained at standard conditions

of temperature and pressure was 7.18 liters and 3.18 liters at the end of run 1 and run 2 respectively. Cumulative methane yield at the standard conditions of temperature and pressure was 0.14 liters of methane per gram wet weight for run 1 and 0.15 liters of methane per gram wet weight for run 2. Daily methane production rate peaked at $0.245 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ for mixed digester on day 40 in run 1 and $0.195 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ for run 2 on day 2. The daily methane production rate dropped to $0.008 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ and $0.011 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ for run 1 and run 2 respectively at the end of the run. Run 1 required 34 days to achieve 95% of its methane yield potential while run 2 required 7 days for the same.

The results highlights the direct correlation between the amounts of biogas produced to the amount of substrate degraded which validates the mass balance of the process. The pH of all the runs were in the range of 7.0- 8.0. The validation of mass balance and the pH range justifies the validation of the biogasification and a stable digestion.

The profile of SCOD for run 1 is shown in Figure 5-4. SCOD concentration initially accumulated reached a maximum and decreased to minimum around 4.5 g L^{-1} . The digesters in run 1 showed significantly higher SCOD than other runs due to the use of inoculum with high SCOD. The higher degradation rate of SCOD in run 1 was consistent with the higher daily methane production rate seen in Figure 5-4. Run 2 started with low initial SCOD. As the SCOD concentration decreases, cumulative methane yield increases accordingly.

5.5 Conclusions

NASA Lunar waste stream was biogasified in single-stage, batch anaerobic digesters under mixed and unmixed condition respectively for 2 runs. The digester at bulking condition produced 11.43% and 50% more methane than the digester at mixing

condition in run 1 and 2 respectively after the same digestion time. In addition, the digester at unmixed condition also exhibited higher organic matter degradation rate. Unmixed system seems to do better than mixed system for NASA Lunar waste stream.

Biogasification of Bio Bag suggests that adopted inoculum gives better methane yield than the fresh culture.

Table 5-1. Single Stage Biogasification of Lunar Wastes

S N	Sample	Dry Weight (g)	Working Volume (L)	Total Methane @ STP (L)	Gas Analysis	Solids Analysis		Liquid Analysis		Duration to produce 95% methane yield potential (Days)	Mass of Biogas recovered (g)
					Cumulative CH ₄ Yield (L/g) @STP	Degradation (g)	Degradation (%)	Residual SCOD (g/L)	Final pH		
1	Mixed-1	100	4.0	1.76	0.035	44.80	44.80%	8.40	7.20	6	4.71
2	Mixed-2	25	2.0	3.12	0.12	14.30	57.20%	7.84	8.66	16	8.36
3	Unmixed-1	100	4.0	3.46	0.039	42.00	42.00%	9.12	7.53	5	9.27
4	Unmixed-2	25	2.0	4.55	0.18	14.10	56.40%	8.10	8.86	24	12.19

Table 5-2. Biogasification of Bio-Bag

S N	Sample	Dry Weight (g)	Duration (Days)	Total Methane (L)	Gas Analysis	Solids Analysis		Liquid Analysis		Duration to produce 95% methane yield potential (Days)	Mass of Biogas recovered (g)
					Cumulative CH ₄ Yield (L/g) @STP	Degradation (g)	Degradation (%)	Residual SCOD (g/L)	Final pH		
1	Run-1	50	64	7.18	0.14	32	64%	5.90	8.53	34	19.23
2	Run-2	20	70	6.10	0.15	45	90%	4.54	8.59	63	19.00

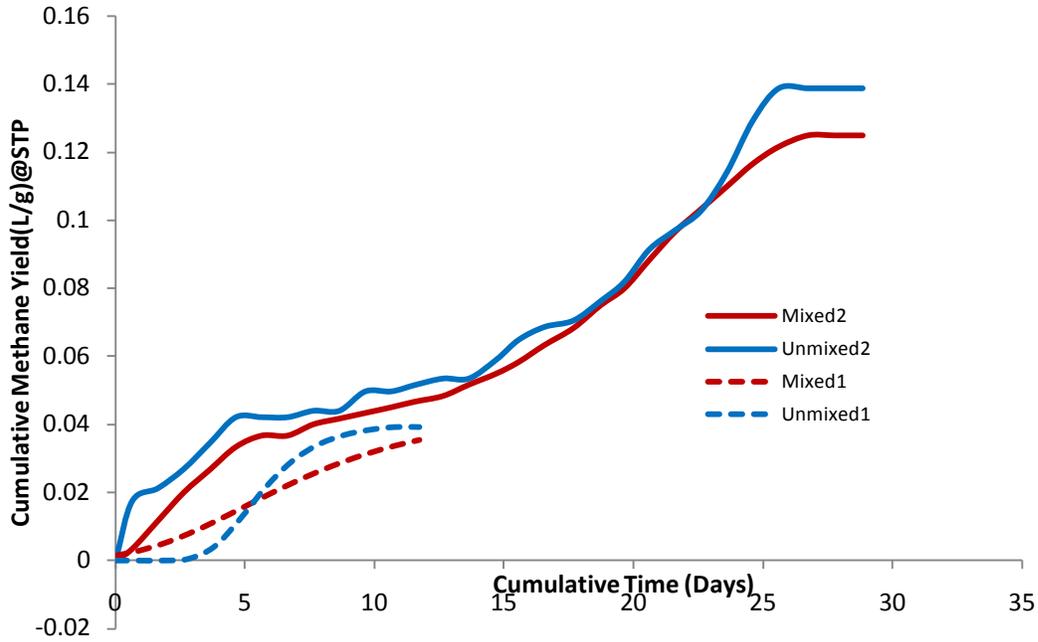


Figure 5-1. Comparison of Mixed and Unmixed System for Lunar Waste Biogasification

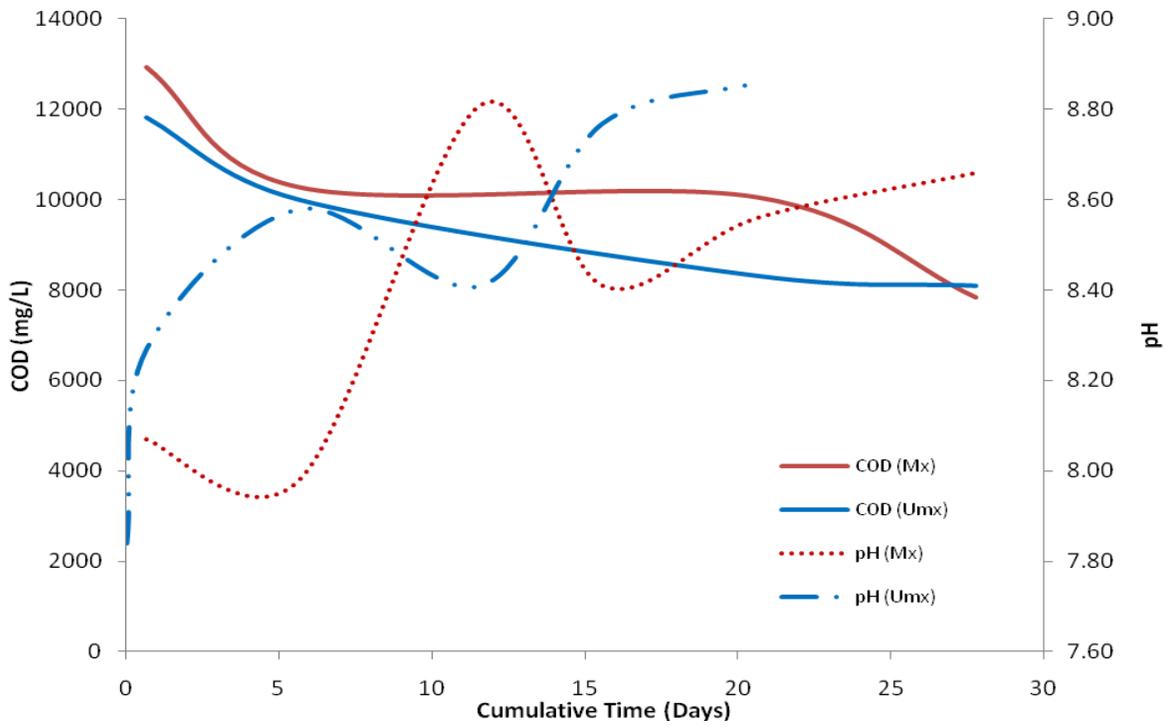


Figure 5-2. COD/pH variations in Mixed and Unmixed System

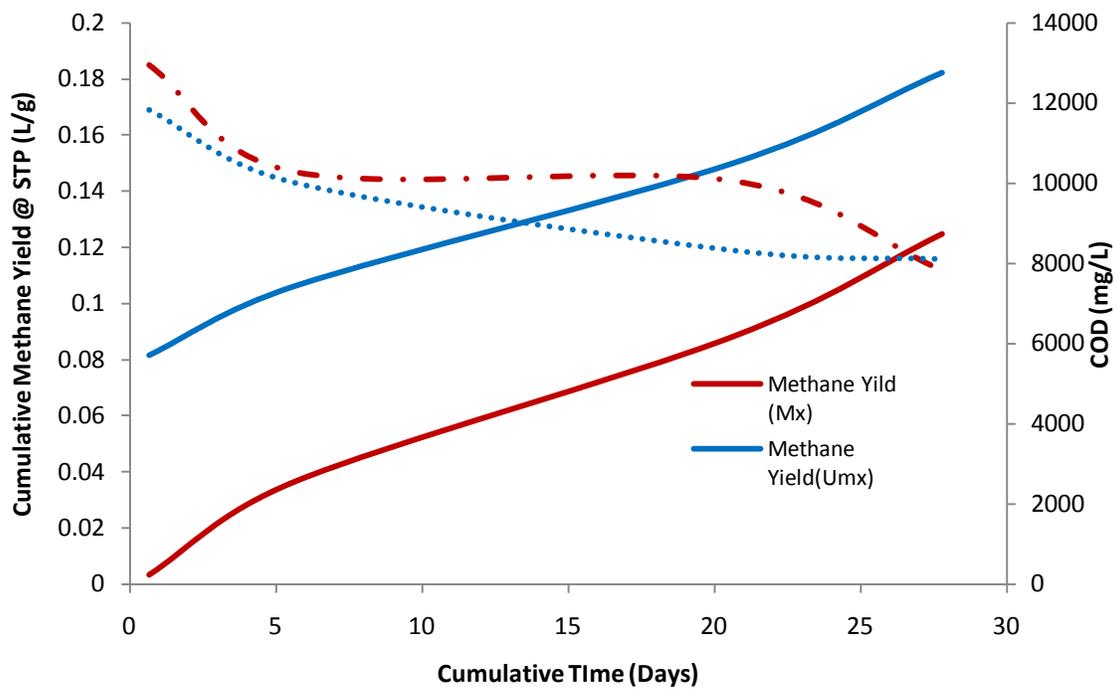


Figure 5-3. Methane Yield/COD variations in Mixed and Unmixed System

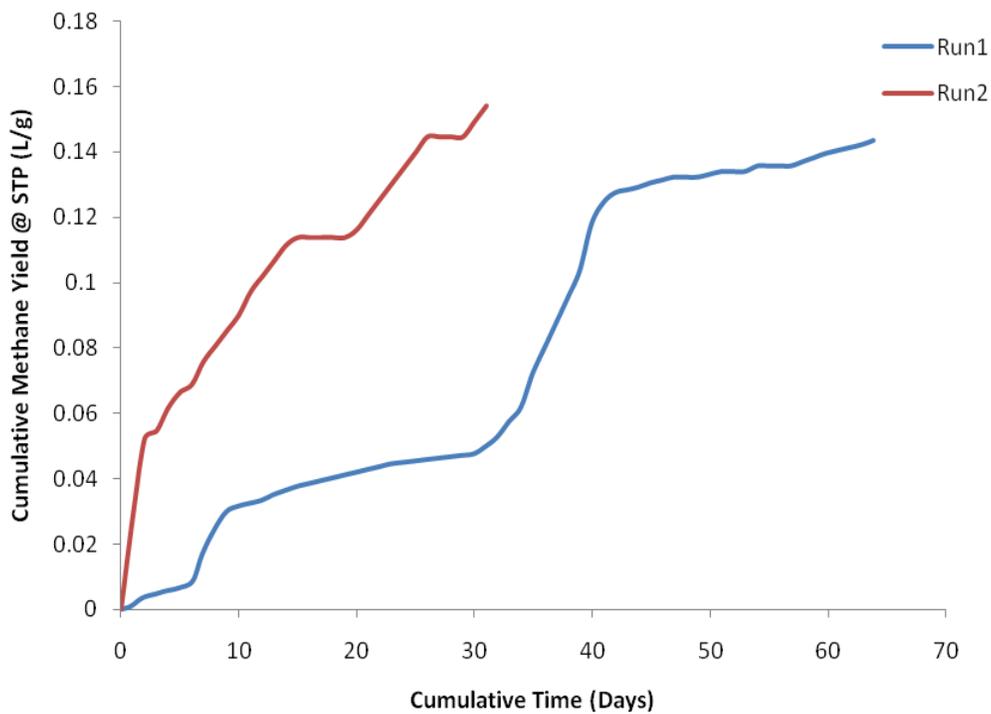


Figure 5-4. Cumulative Methane Yield for Biogasification of Bio Bag

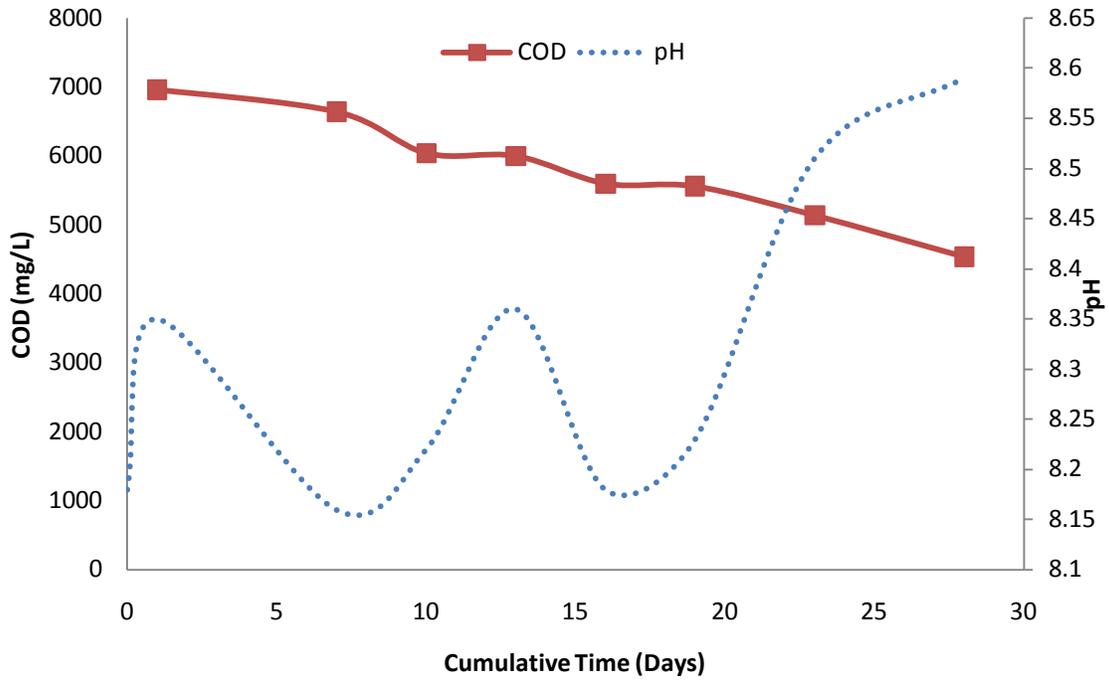


Figure 5-5. COD/pH Variations in the Biogasification of Bio Bag

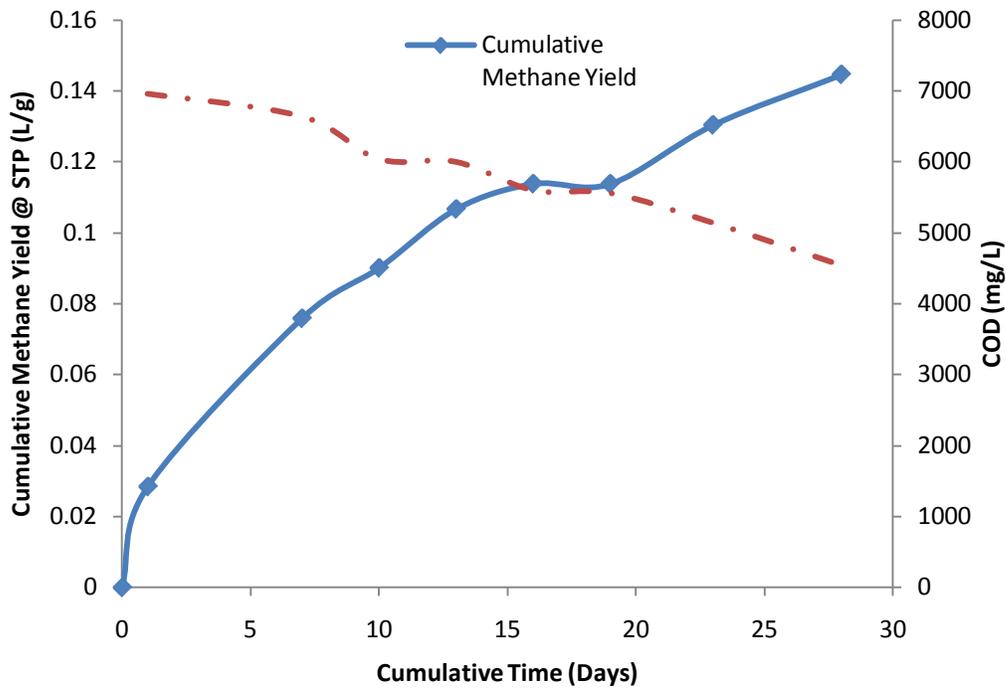
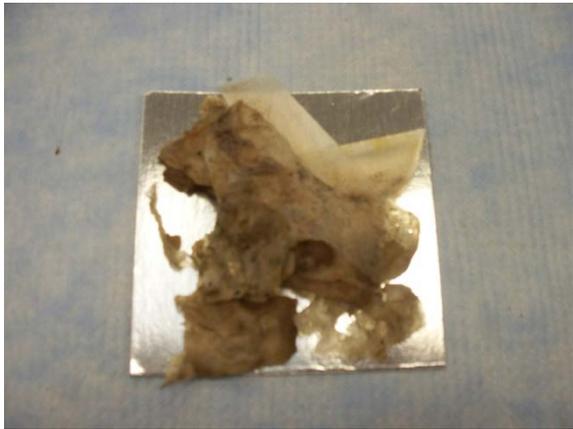


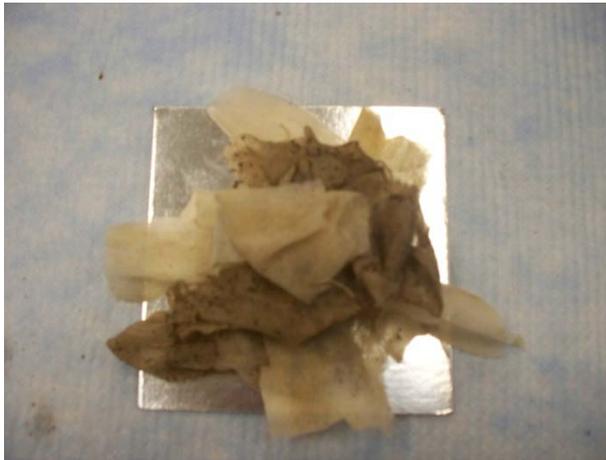
Figure 5-6. Methane Yield/COD Variations in the Biogasification of Bio Bag



A



B



C



D

Figure 5-7. Degraded samples from single stage biogasification of Lunar wastes : A) Packaging material B) Clothes C) Wipes D) Grey Tape



Figure 5-8. Degraded Bio Bag from single stage biogasification

CHAPTER 6 TWO STAGE BIOGASIFICATION STUDIES AND CONCEPTUAL DESIGN

6.1 Introduction

This part of the study demonstrates two stage biogasification of NASA Lunar wastes. Two stage operation was carried out, one of which acted as a main digester having 2 L working volume into which the waste was loaded and the other was a mixed digester having a 3.5 L working volume to digest soluble organic matter produced in the first stage. About 600 ml of liquid was transferred from first stage to second and vice versa daily. The progression of an Experiment was measured by the evolution of methane with time. After the feedstock was degraded the reactor was opened and the residue removed and analyzed. The aim of this study was to demonstrate an appropriate digester design that could be used for efficiently biogasifying Lunar mission waste. From this information a prototype digester was designed sized for a four-person crew on a one year exploratory Lunar space mission. Research presented here supports the use of high-solids anaerobic digestion for bioregenerative purposes and stabilization of the organic components of solid wastes during extended Lunar space missions.

6.2 Two Stage Biogasification Studies

6.2.1 Background

Typically, when a two stage system is used, the first one harbors the liquefaction-acidification reactions, with a rate limited by the hydrolysis of cellulose, and the second one harbors the acetogenesis and methanogenesis, with a rate limited by the slow microbial growth rate (Liu and Ghosh, 1997; Palmowski and Miiller, 1999). With these two steps occurring in distinct reactors, it becomes possible to increase the rate of

methanogenesis by designing the second reactor with a biomass retention scheme or other means (Weiland, 1992; Kiibler and Wild, 1992). In parallel, it is possible to increase the rate of hydrolysis in the first stage by using microaerophilic conditions or other means (Capela et al., 1999; Wellinger et al., 1999). The application of these principles has led to a great variety of two-stage designs. However, in the hybrid two-stage system employed in this study, methanogenesis occurs in both digesters. The purpose of the second stage is to degrade readily solubilized organic matter from the first stage and convert it to methane. It was found that waste generated in a lunar mission contains a large fraction of readily solubilizable organic matter. If this is not quickly removed from the first stage, it has the potential to acidify rapidly, causing the pH to drop and inhibiting further degradation. The design for second stage of the system could be any one of the many designs commonly used for anaerobically digesting wastewater. For simplicity a stirred tank digester was used in this study.

6.2.2 Results and Discussion

The profile of cumulative methane potential of NASA Lunar waste in two stage system is shown in Figure 6-6. Run1 started with 25 g of the waste gave total 5.56 L of methane in 18 days out of which 2.08 L generated from the first stage and 3.48 L generated from the second stage. Total cumulative methane yield for run1 was 0.200 liters of methane per gram of waste at the standard condition of temperature and pressure. Run2 was started with 50 g of the waste gave total 5.70 L of methane in 8 days out of which 3.79 L generated from the first stage and 1.95 L generated from the second stage. Total cumulative methane yield for run1 was 0.210 liters of methane per gram of waste at the standard condition of temperature and pressure. It is interesting to note that run2 achieved the same cumulative methane yield as run1 in less time.

Duration for run2 was 10 days less than run1. This could be because of the adaptation of microbial consortia in the digester to the substrates in the waste stream during run1.

Daily methane production rate peaked at $0.239 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ for digester on day 2 in run 1 and $0.735 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ for run2 on day 2. The daily methane production rate dropped to $0.017 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ and $0.051 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ for run 1 and run 2 respectively at the end of the run. Run 1 required 15 days to achieve 95% of its methane yield potential while run 2 required 6 days for the same.

The results highlights the direct correlation between the amounts of biogas produced to the amount of substrate degraded which validates the mass balance of the process. The pH of all the runs were maintained in the range of 7.0- 8.0 without addition of any chemicals indicating stable digestion.

The profile of SCOD for run 1 is shown in Figure 6-9. SCOD concentration initially accumulated reached a maximum to and decreased to minimum around 5.5g/L. The digesters in run2 showed significantly higher SCOD of 16.8 g/L because of the larger quantity of waste loaded into the digester. The higher degradation rate of SCOD in run 2 was consistent with the higher daily methane production rate seen in Figure 6-7. Run 1 started with low initial SCOD of 10.2 g/L.

The rate of degradation in the two stage system is higher than one stage system discussed in Chapter 5. For the same amount of feedstock, two stage system achieved the methane potential 55.55% faster than the one stage system. About 6% more solids degradation was achieved in two stage system compared to single stage system. It was

possible to apply a higher organic loading of 25 g/L in the two stage system as compared to 12.5 g/L in one stage.

6.3 Full Scale Conceptual Design

6.3.1 Background

For lunar applications, a two stage system was envisioned. The system was designed so that feed would be collected, coarsely shredded, mixed with recycled digester effluent to give the desired concentration of solids, and compacted to a density of 200 kg/m³. The digestion time was forecasted to be 8 days per batch. The energy potential for one year exploratory lunar mission was calculated based on the laboratory scale experimental data. Biogas from anaerobic digester would be treated to recover carbon dioxide and remove hydrogen sulfide and other contaminants. The methane could be used as fuel. The residue from the digester was readily dewatered as it contained only non degradable plastic material. Pathogens would also be inactivated during the anaerobic process as the digestion was carried out at thermophilic conditions (Bendixen, 1994; Engeli, 1993).

6.3.2 Reactor Volume Calculations

The expected amount of waste entering the anaerobic digestion system would be that generated by 4 person crew. For typical 8-days anaerobic digestion cycle as demonstrated in two stage work above each reactor would contain 8-days worth of solid waste. Individual lunar waste components add to 1.101 kg/(CM-D). Considering the crew of 4 astronauts, the amount of waste generated will be $1.101 \times 4 = 4.404$ kg/D.

Over 8 days, 35.23 kg ($4.404 \times 8 = 35.23$) of waste would be generated. This would be collected in one vessel over 8 days.

After the dry waste is placed in the digester and compacted to 200 kg/m^3 , recycled digester effluent must be added to initiate anaerobic digestion. As demonstrated above for the two stage system, for 50 g of the Lunar waste, 2.00 L of inoculum was added. Hence, to digest 35.23 kg of the waste it would require 1.5863 m^3 of liquid (or recycled digested effluent) for a total volume of 1.41 m^3 .

Assuming the reactor is cylindrical-shaped and assuming no headspace (due to low gravity application). The dimensions of the reactor can be calculated as follows:

$$V = \pi \times R^2 \times H \quad [6-1]$$

If the radii of the vessel is 0.502 m (R), the height would be 1.78 m (H) for a volume of $V_P = 1.41 \text{ m}^3$

6.3.4 Sizing the second stage of two stage system

Upon addition of liquid into digester the readily solubilized organic matter leaches out and within a few hours the soluble organic content increases to 16.8 g COD/L.

In experiments here the volume of second stage was 87.5% more than the first stage but as noted earlier the second stage design was not optimal. Under optimized conditions an anaerobic filter (the design employed for second stage) can be sized to handle 8 g COD/L/day (Lee et al. 2009). Assuming that as in the experiments if liquid volume equal to 30% of total volume of first digester is transferred to second stage every 24 hours, the size of the second stage works out to be 0.846 m^3 .

The volume of the anaerobic filter = 0.846 m^3

R = 0.502 m

H = 1.0686 m

Surface Area= 4.9539 m²

6.3.5 Digester Operations

6.3.5.1 Thermophilic System

The Digester-1 will be filled with the solid wastes and will be operated for 8 days. Simultaneously, the Digester-2 will act as the trash collector during these 8 days. At the end of digestion in Digester-1, liquid from Digester-1 will be transferred to Digester-2 initiating the digestion process in Digester-2. Digester-1 will be emptied, and will act as trash collector. This process will be repeated. A total of 46 batches of waste will be treated over a year.

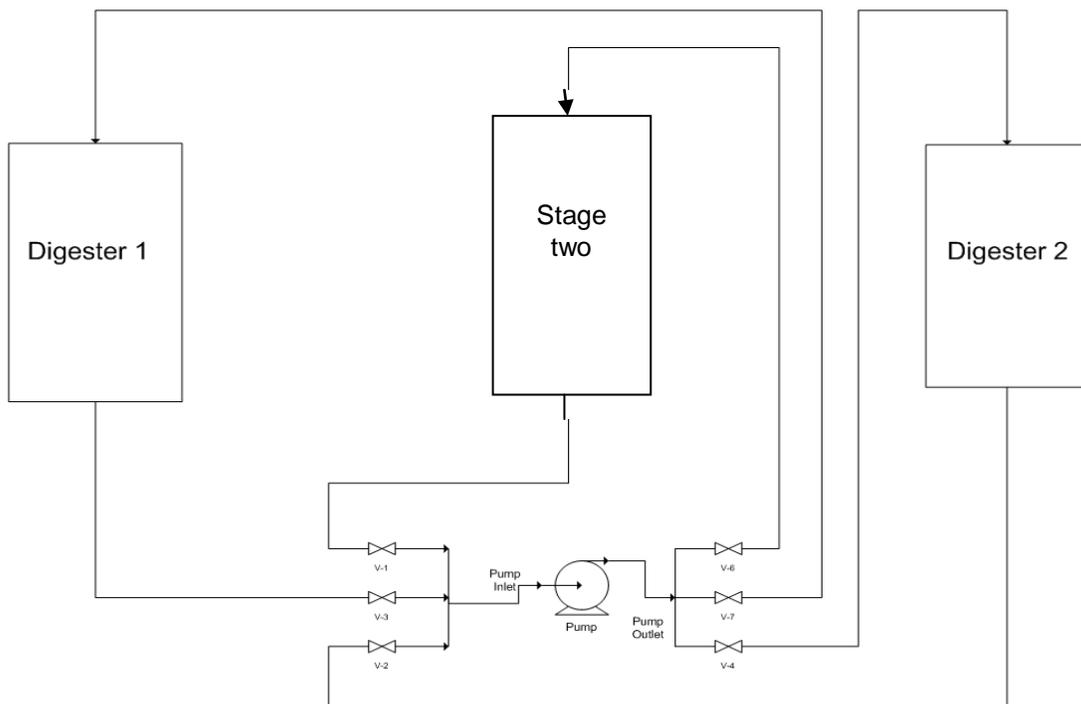


Figure 6-1. Thermophilic Digester Operation

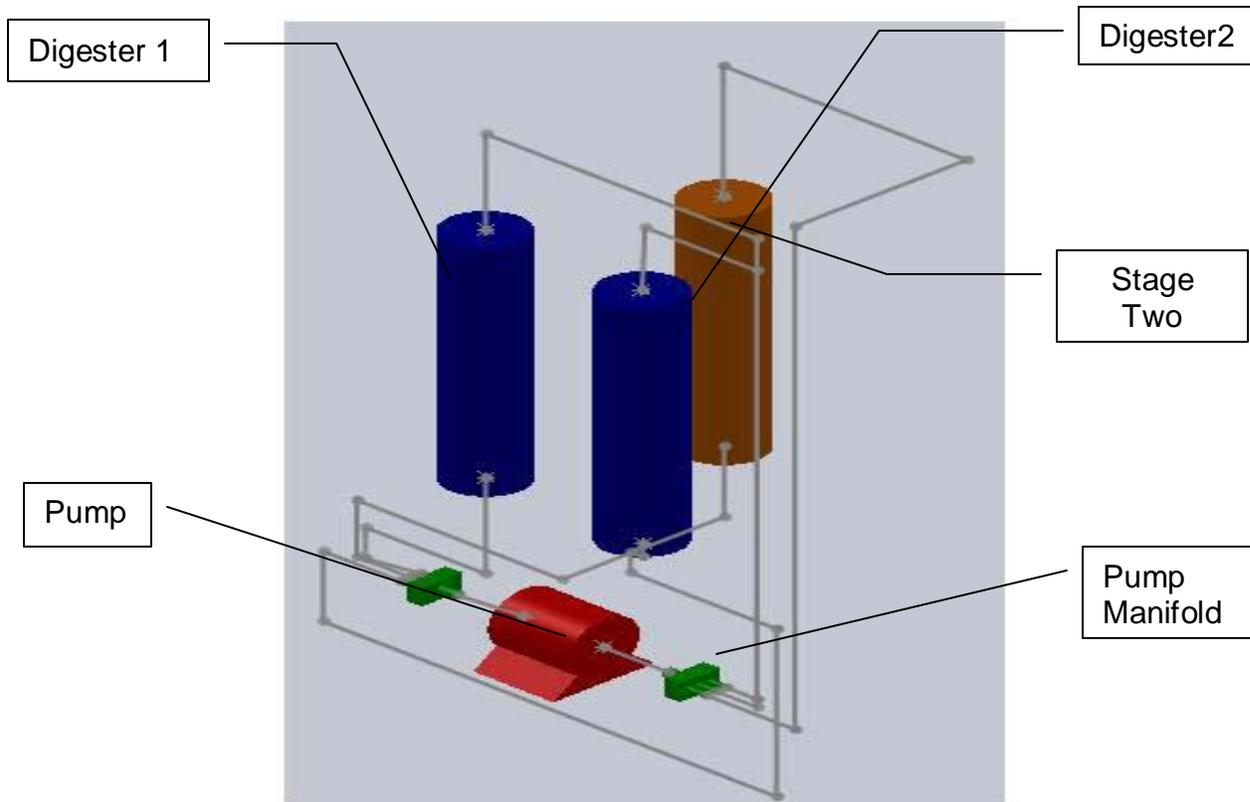


Figure 6-2. NASA Lunar Thermophilic Digesters: 3 dimensional view

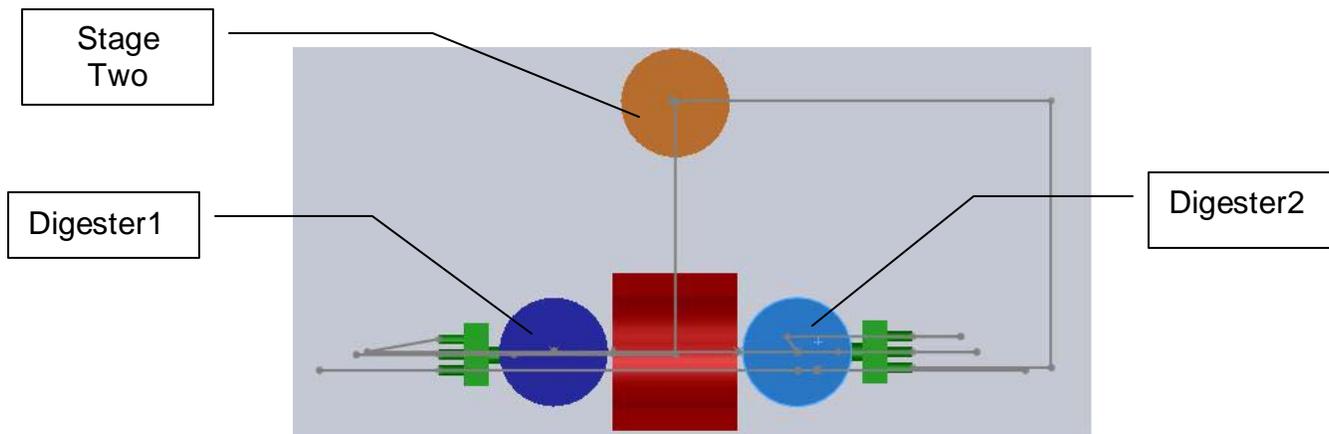


Figure 6-3. NASA Lunar Thermophilic Digesters: Top view

6.3.5.2 Mesophilic System

It was seen that the rate of degradation under mesophilic conditions is half as that achieved under thermophilic conditions. Therefore, period of digestion of a batch of

waste will be 16 days and 3 digesters will be required if similar vessel volumes as that used for thermophilic digestion is employed. Digester-1 will be filled with the solid wastes over eight days and operated for 16 days. From day 9, Digester-2 will act as the trash collector for the next eight days. On day 17 the process will be initiated in digester-2 and digester -3 will now serve as trash collector for the next 8 days. By day 24, Digester-1 will become available for trash collection. In total the vessels will be loaded 46 times. The anaerobic filter (or second stage) will be required to treat liquid streams from two digesters. Since typical loading rates for anaerobic filter under mesophilic conditions are half as that for thermophilic digester, the volume requirements will be four-fold for this vessel, i.e. 3.384 m³.

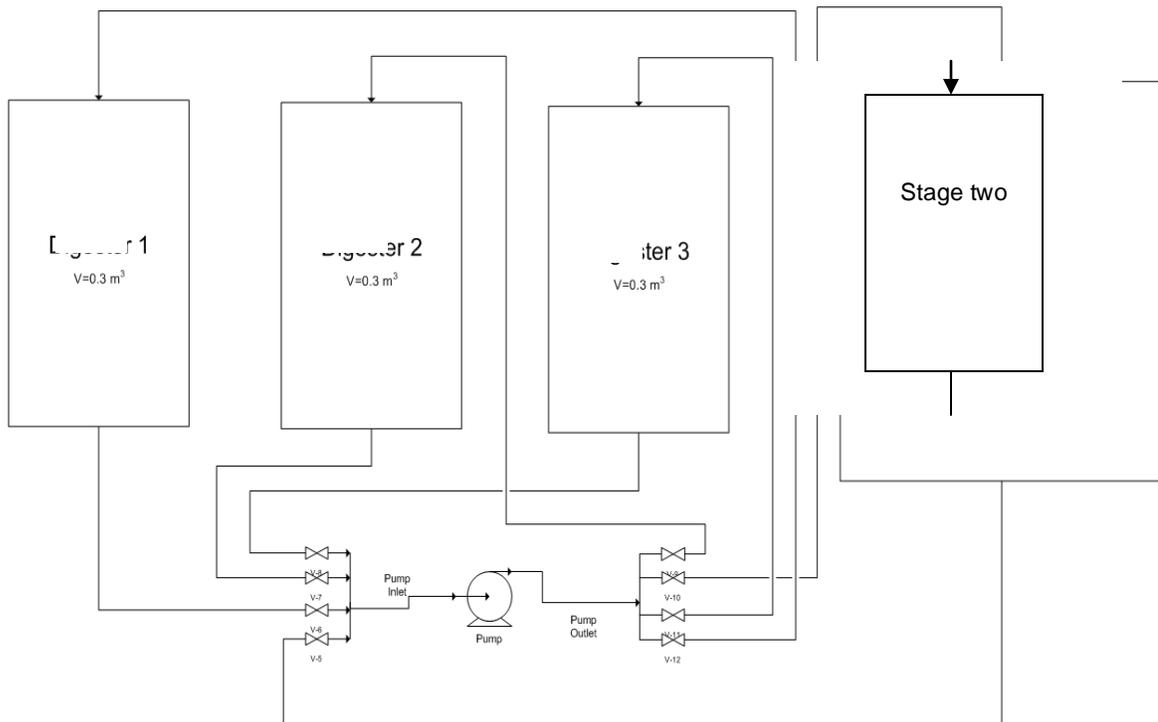


Figure 6-4. Mesophilic Digester Operation

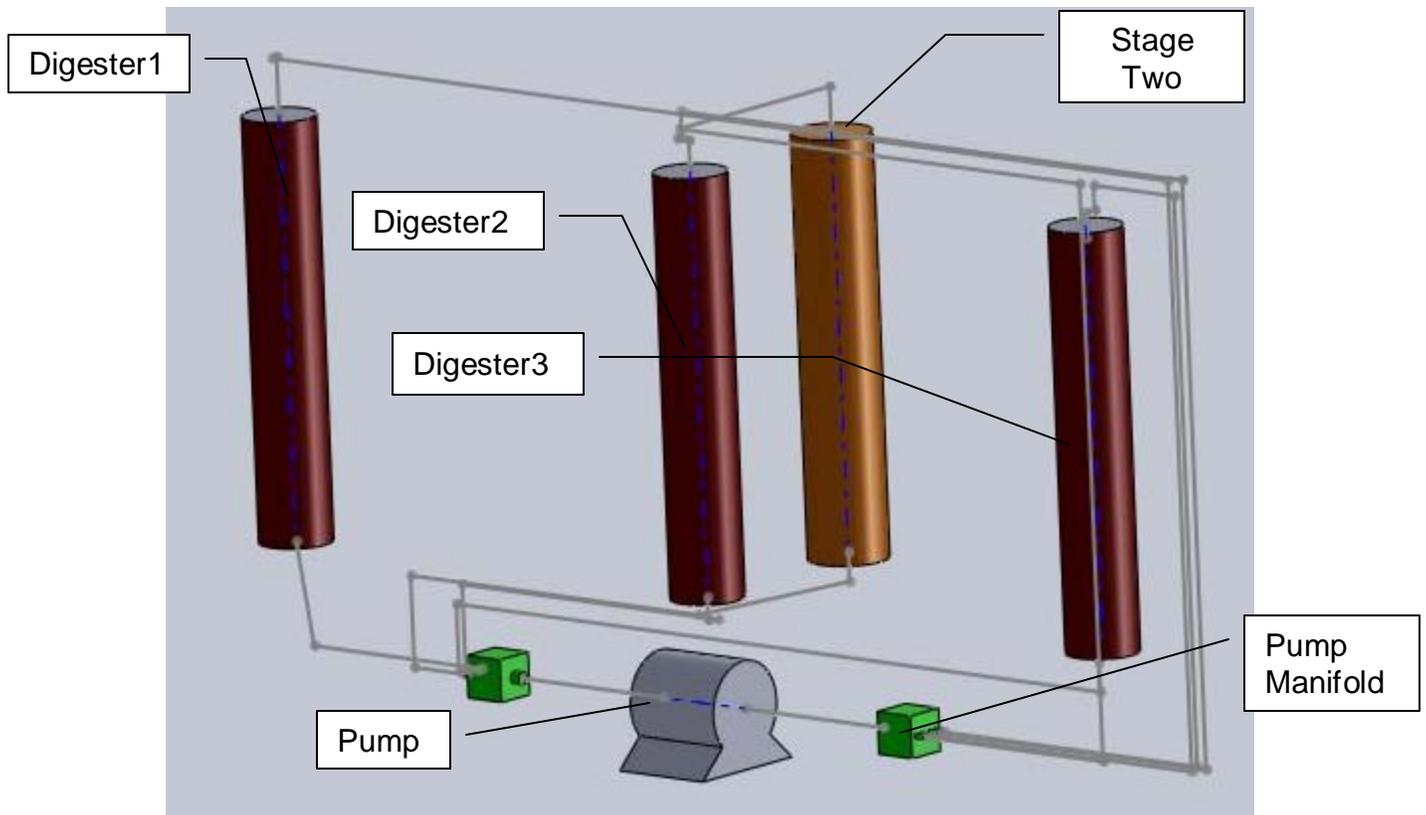


Figure 6-5. NASA Lunar Mesophilic Digesters: 3 dimensional view

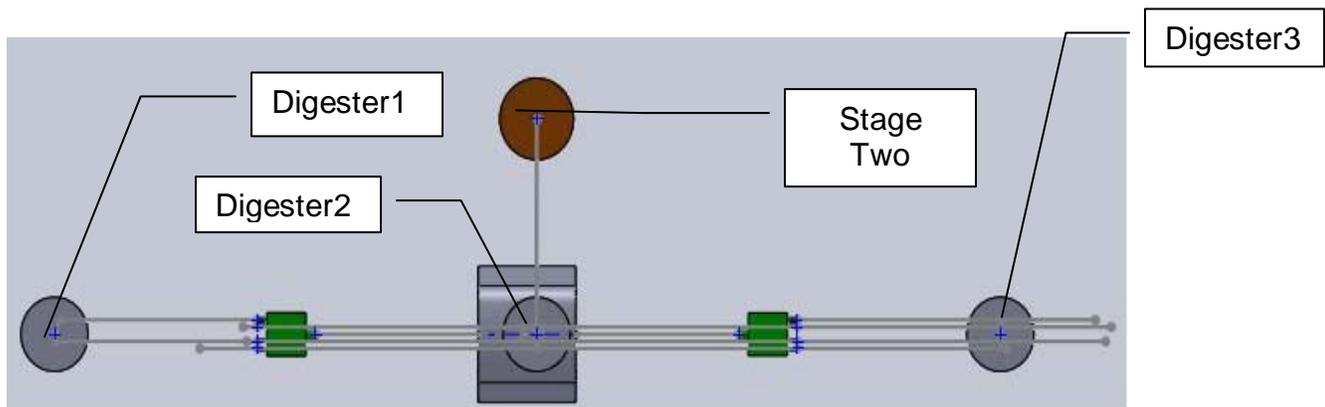


Figure 6-6. NASA Lunar Mesophilic Digesters: Top view

6.4 Energy Potential of Anaerobic Digestion Operations During 1 Year Exploratory Lunar Space Mission

Experimental Methane Potential = 200 L/(CM-D)

For a crew of 4 astronauts= 200 * 4 = 800 L/D/kg

Total methane potential = $800 * 1.101 = 880.8 \text{ L/D}$

For a Batch of 8- Days = $880.8 * 8 = 7046.4 \text{ L} = 7.046 \text{ m}^3$

Calorific Value of Methane = $37,000 \text{ KJ/m}^3$

Total Energy potential of Lunar Wastes = $260,716 \text{ KJ}$ for a batch of 8 days

For a 1 year exploratory lunar space mission, there will be 46 such batch digestions.

Energy potential for 1 year Lunar Wastes = $11,992,972 \text{ KJ}$ which is almost 12 M KJ .

In the units of power = 0.3824 kW

6.5 Energy Requirements

6.5.1 Energy Required for the Digester Start-Up

The amount of heat needed to heat a subject from one temperature level to another can be expressed as:

$$Q = c_p m \Delta T \quad [6-2]$$

where

Q = amount of heat (kJ)

c_p = specific heat capacity (kJ/kg.K)

m = mass (kg)

ΔT = temperature difference between hot and cold side (K)

For Thermophilic (55 C) Operation:

$$Q = 4.19 \text{ (KJ/kg.K)} * (1410 + 1410 + 846) \text{ kg} * (328 - 298) \text{ K}$$

$Q = 460,816 \text{ KJ}$ for start up

For Mesophilic (38 C) Operation:

$$Q = 4.19 \text{ (KJ/kg.K)} * (1410 * 3 + 3384) \text{ kg} * (311 - 298) \text{ K}$$

$Q = 414,735 \text{ KJ}$ for start up

6.5.2 Heat Losses from Insulation

(Source: Thermalinc website, <http://www.thermalinc.com/math/insulation.htm>, accessed on 10/25/09)

For Thermophilic (55 C) Operation:

Required heat for maintaining the temperature in the tank= $0.1656 \text{ W/m}^2\text{C}$

$$= 0.1656 * 17.7661 * 30$$

$$= 88.2366 \text{ W}$$

$$= 2,782,631 \text{ KJ per year}$$

For Mesophilic (38 C) Operation:

Required heat for maintaining the temperature in the tank= $0.1656 \text{ W/m}^2\text{C}$

$$= 0.1656 * 34.2838 * 13$$

$$= 73.8062 \text{ W}$$

$$= 2,327,551 \text{ KJ}$$

6.5.3 Heat of Vaporization

For Thermophilic (55 C) Operation:

Biogas moles for a batch=524.28 moles

Water moles=12.5% Biogas Moles

$$G = 0.125 G + 524.28$$

$$\text{Heat of Vaporization} = 2370.8 * 1.34 = 3176.87 \text{ KJ per Batch}$$

For Mesophilic (38 C) Operation:

Biogas moles for a batch=524.28 moles

Water moles=6.54% Biogas Moles

$$G=0.0654 G + 629.14$$

$$\text{Heat of Vaporization} = 2411.7 * 0.79 = 1911.15 \text{ KJ per Batch}$$

6.5.4 Energy Requirement of Pump

To recirculate the leachate within the digester and to transfer liquid between stage 1 and stage 2 a pump must be used. As calculated earlier, for one digester, the required leachate volume is 1.413 m^3 . It is assumed that the total leachate recirculation/transferred volume per day is 10 times the leachate volume in digester, i.e., 14.13 m^3 and the pumps work 20 minutes per 2 hours. So, the leachate recirculation flow rate is:

$$Q = 10 * 1.413 (\text{m}^3/\text{d}) / (24/2) * 20 (\text{min/d}) = 0.0588\text{m}^3/\text{min}$$

According to Energy Conservation Law, the energy required can be calculated as follows:

$$E_T = 0.5 m v^2 + m g H \quad [6-3]$$

$$v = Q/D^2 \quad [6-4]$$

Hence, Pump energy = 51,038 J per day

So, its 18,629 KJ per year for Thermophilic (55 C). It will be twice this for mesophilic system as liquid needs to be recirculated between two digesters and anaerobic filter.

Tables 6-1 and 6-2 summarizes the energy consumption for operation of thermophilic and mesophilic digester systems. The net energy gain is 72% and 76% for thermophilic and mesophilic operation respectively. Most of the energy is consumed for maintaining the temperature in the vessels. If temperature losses can be minimized with better insulation then net energy gain would be even higher. Table 6-3 lists the

initial amount of water required to start up the digestion process. Due to the larger volumes mesophilic operation requires more water. After start up the effluent from digester will be recycled and no further make up water will be required. Taking into consideration the increased water requirement for mesophilic operation and given that excess net energy gain is small, thermophilic operation is recommended. More thermophilic operation has the added advantage of higher levels of pathogen inactivation.

Table 6-1. Energy Consumption for Lunar Digesters

S.N.	Type of Energy	Thermophilic (55 C) System (KJ)	Mesophilic (38 C) System (KJ)
1	Energy for digester start up	460,816	414,735
2	Energy to compensate heat losses from insulation	2,782,631	2,327,551
3	Energy to compensate heat of vaporization	146,136	87,913
4	Energy consumed for pumping operation	18,629	37,258
Total		3,408,212	2,867,457

Table 6-2. Net Energy Gain for Lunar Digesters

S.N.	Type of Energy	Thermophilic (55 C) System (KJ)	Mesophilic (38 C) System (KJ)
1	Energy Potential of Lunar Wastes	11,992,972	11,992,972
2	Energy Consumption during digestion operation	3,408,212	2,867,457
Net Gain		8,584,760	9,125,515

Table 6-3. Initial water requirement for Lunar Digesters

S.N.	Water Requirement	Thermophilic (55 C) System (L)	Mesophilic (38 C) System (L)
1	Water requirement for the digesters	2820	4,230
2	Water requirement for the anaerobic filter	846	3,384
TOTAL		3,666	7,614

6.6 Comparison of Lunar mission wastes digesters with Mars mission wastes

Previously Haley et al. (2002) developed a design for high solids anaerobic digestion of wastes generated during long term Mars mission. The wastes generated during a long term Mars mission are quite different from that generated in a Lunar base.

The composition of Mars mission waste is as follows:

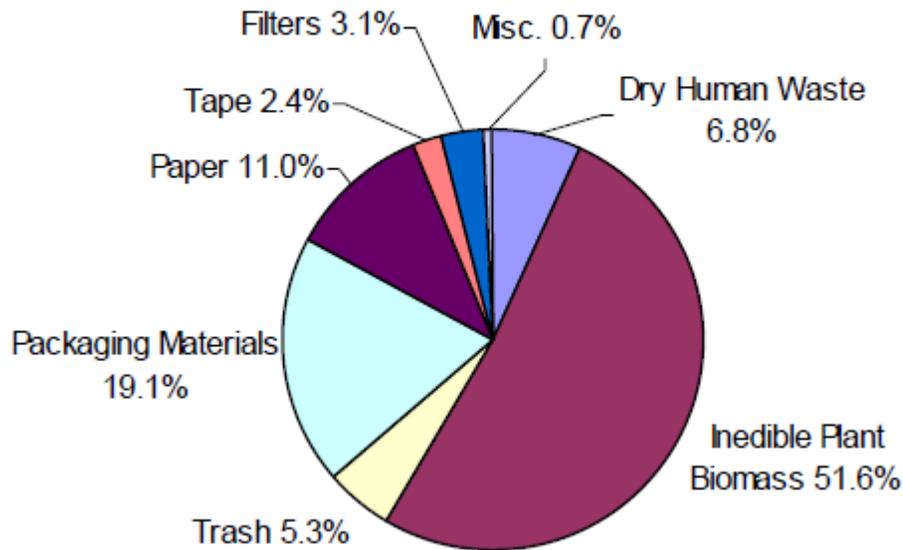


Figure 6-7. Space Mission Waste Composition (source: Haley et al, 2002)

The daily solid waste streams for a 6-person crew during a 600-day exploratory mission to Mars were estimated as follows:

Table 6-4. Estimates of daily solid waste stream for Mars mission (source: Haley et al., 2002)

Waste Component	Dry Wt., Kg	Percent of total	Ash%	Organic Matter, Kg	Moisture, %
Dry human waste	0.72	9.4	5	0.68	85
Inedible plant biomass	5.45	51.4	5	5.2	75
Trash	0.56	5.3	5	0.53	10
Paper	1.16	10.9	5	1.1	10
Packaging materials	2.02	19.0			
Tape	0.25	2.4			
Filters	0.33	3.1			
Misc.	0.07	0.7			
Total	10.6	100		7.5	

For these wastes Haley et al. (2002) had developed a 5-vessel (used as trash collectors and digesters) and 2-reservoir system with the volume of each digester equal to 0.125 m³ while that of reservoirs was 0.062 m³. This system handles 7.5 kg of waste/day in a batch of 5 days. The total size of the reactors for Mars mission wastes reported by Haley et al. (2002) was 0.749 m³ to treat 37.5 kg of waste per batch. While, for treating a batch of 35.23 kg of Lunar mission wastes, the total size of the reactors was estimated to be 4.94 m³. The difference in sizes is mainly because of the difference in the composition of waste streams for Lunar mission and Mars mission.

Lunar mission waste components, as reported in Table 3-1, has 61.30% of the organic matter of the waste stream, while Mars mission waste components has 70.75% of the organic matter of the total waste stream. Human wastes, adhered food, uneaten food form the soluble part of the organic waste component which is 40% of total organic matter present. While, Mars mission waste has only human wastes as the soluble part

of the organic waste which forms 9% of total organic matter present. As Lunar mission wastes has more soluble organic matter compared to Mars mission wastes, the leachate turns acidic quickly upon water addition. To handle this acidic leachate a separate anaerobic filter digester is required. This was not a necessity for wastes generated in Mars as most of the organic component is plant biomass which does not solubilize quickly. Hence the volume of the digester is more for the Lunar mission wastes compared to Mars mission wastes.

6.7 Conclusion

The conceptual design for the NASA Lunar waste digesters has been developed based on results from pilot scale studies. Thermophilic system seems to do better than mesophilic in terms of mass of the equipment and water requirement. Thermophilic system requires 50% less water than mesophilic system which will be an important consideration for long term space mission. Mesophilic system does slightly better in terms of net energy gain, producing 4% more. Both energy and water are the crucial entities for the long duration space missions, but in terms of cost of transportations of equipment and reactors, thermophilic system should be a good choice.

Table 6-5. Two stage biogasification of Lunar wastes

S . N .	Sample	Dry Weight (g)	Duration (Days)	Total CH4 (L)	CH4 from stage1	CH4 from stage2	Gas Analysis	Solids Analysis		Liquid Analysis		Duration to produce 95% methane yield potential (Days)	Mass of Biogas recovered (g)
							Cumulative CH4 Yield (L/g) @STP	Degradation (g)	Degradation (%)	Residual SCOD (g/L)	Final pH		
1	Run-1	25	18	5.56	2.08	3.48	0.200	15.1	60.40	5.55	8.23	15	14.89
2	Run-2	50	8	5.70	3.79	1.95	0.210	33.5	67.00	3.12	7.97	6	15.27

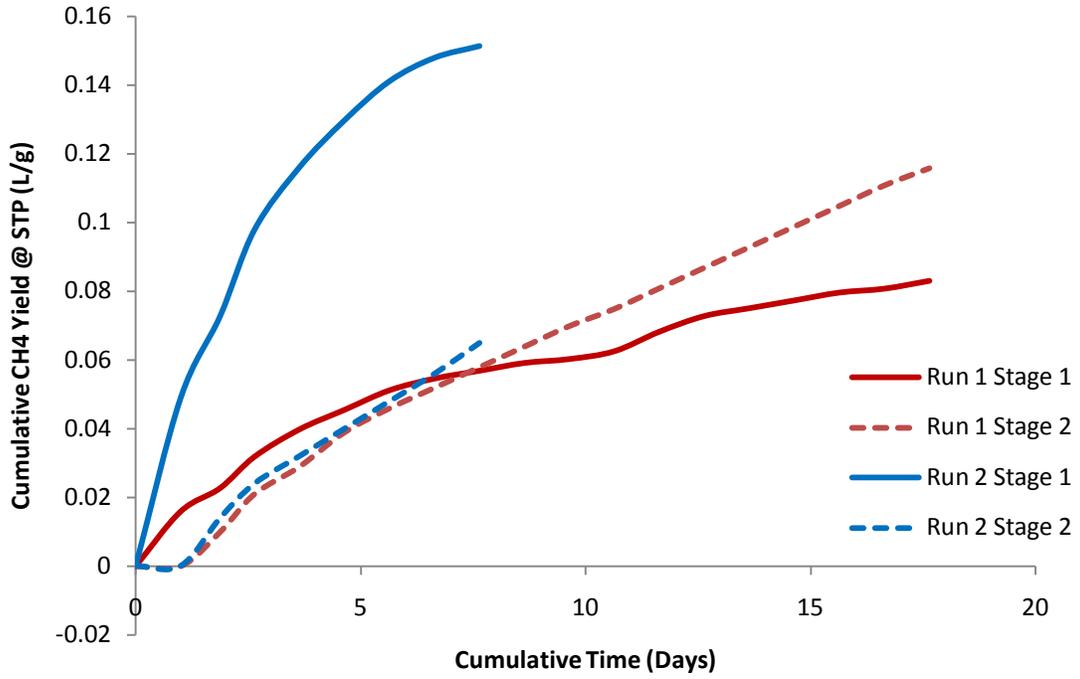


Figure 6-8. Cumulative Methane Potential of Two Stage System

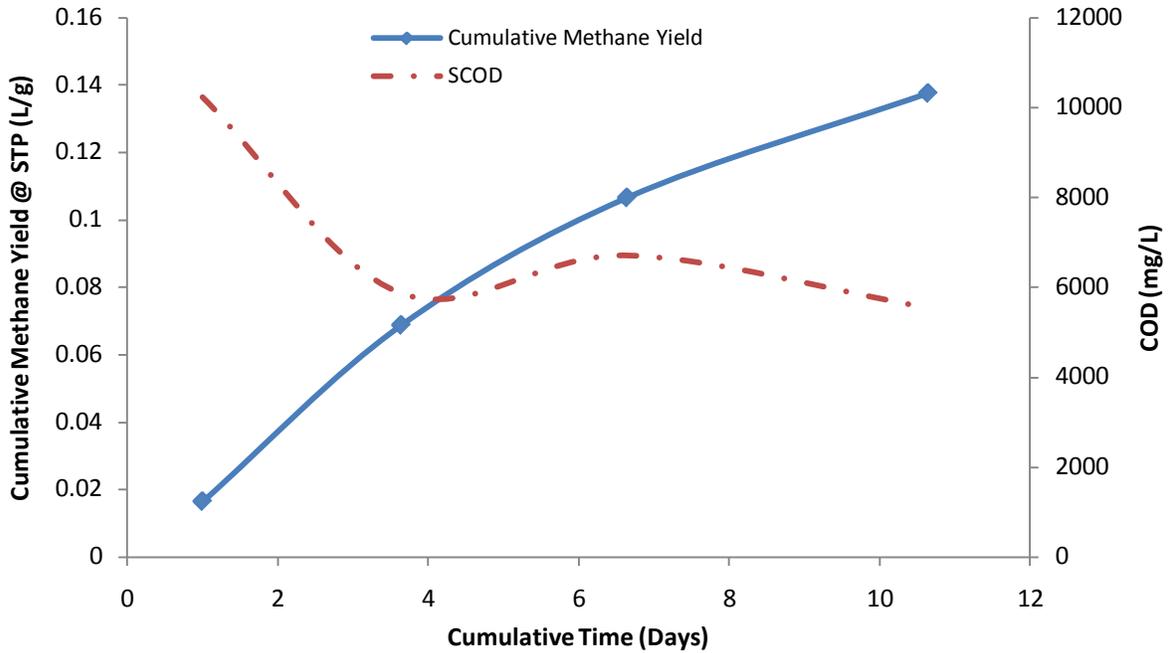


Figure 6-9. Variation of cumulative methane yield and SCOD in digester for two stage system (Run-1)

CHAPTER 7 CONCLUSIONS AND FUTURE WORK

7.1 Conclusions

Research presented here supports the use of high-solids leachbed anaerobic digestion for bioregenerative reduction and stabilization of the organic components of solid wastes during one year exploratory Lunar space missions. Initial biochemical methane potential studies and one stage as well as two stage studies followed by conceptual prototype reactor have shown positive results for decreased retention time and increased reduction of biomass in the modified anaerobic digestion system. The critical findings in the study are as follows:

1. Human waste and food waste demonstrated highest methane potential of 0.856 and 0.481 L/g at standard condition of temperature and pressure (STP) and complete biodegradability. Paper, cotton and clothing also showed good methane potentials and biodegradability. Other components such as packaging materials, wipes, grey tapes etc did not degrade much. The steam treatment did not show any significant effect in terms of methane potential on degradation. Solid residues coming out of the digester did not have much methane potential and some alternative methods must be developed to degrade it and recover energy out of it.
2. Another interesting finding was Bio Bag® achieved 0.344 L/g of methane yield under standard condition of temperature and pressure (STP) with 95% degradation in 106 days under anaerobic condition which seem to be in accordance with ASTM D 6400-04 standards for Compostable Plastics. Repeated biogasification of Bio Bag® showed that adapted inoculum gives better methane yield than the fresh culture. The Bio Bag® also

seems to prove its merit in long term space missions for use in advanced life support system (ALS).

3. The unmixed digester at bulking condition produced 11.43% and 50% more methane than the digester at mixing condition in two replicate runs after the same digestion time.

In addition, the digester at unmixed condition also exhibited a higher organic matter degradation rate. A two stage system seemed to work faster and better than a one stage system. For the same amount of feedstock, a two stage system achieved the targeted methane potential 55.55% faster than the one-stage system. In terms of solids degradation, the two stage system degraded 6% more solids than the single stage system. A two stage hybrid design digested 25 g/L of waste in 8 days.

4. Conceptual designs based on thermophilic and mesophilic operations were compared. The thermophilic system required 50% less water than the mesophilic system which will be an important consideration for long term space mission. Mesophilic system seemed to do better in terms of energy potential of the system. Mesophilic system produced 4% more energy than the thermophilic system. Both energy and water are the crucial entities for long duration space missions, but in terms of cost of transportation of equipment and reactors, a thermophilic system should be a good option.

7.2 Future Work

This work was preliminary and should continue so the following topics of interest can be addressed:

1. The residues coming out of the digester has some moisture content which can be air dried within 24 hours. The experiments here suggest that it does not have any more methane potential. NASA should consider thermal treatment on those residues to get the maximum energy out of it.

2. NASA should consider of developing fully degradable materials for Lunar mission including clothing to enhance anaerobic digestion of entire waste.
3. Optimization of two stage operation to get maximum methane yield and highest degradation in optimized HRT and OLR.
4. Nutrient balances for N, P, and K; need to be determined, with identification of their concentrations in feeds and effluent liquid, solid, and gas streams.
5. Improvement of biogas quality including trace contaminants (like hydrogen sulfide, nitrogenous compounds and volatile organic chemicals).
6. A rough estimate of capital and operating costs for the systems

APPENDIX A
 BIOGASIFICATION STUDIES FOR NASA: JOHNSON SPACE CENTER-HOUSTON

COOLER: 1 NASA JSC LANDSCAPE WASTE

Total weight of the sample received= 7.62 kg

Overall Bulk Density= 0.194 kg/L

Average Bulk Density=0.089 KG/L

Table A-1. NASA JSC Landscape waste composition

S.N.	Components	Wt(kg)	Bulk Density (kg/L)	Vol (L)	Wt Fr	Wt%	Vol Fr	Vol%
1	Grass Clippings	0.03	0.03	0.0009	0.029	2.91	0.003	0.33
2	Weeds	0.05	0.056	0.0028	0.049	4.85	0.01	1.04
3	Leaves (Green) Leaves (Brown)-	0.1	0.03	0.003	0.097	9.71	0.011	1.11
4	Dried	0.03	0.032	0.001	0.029	2.91	0.004	0.36
5	Trimmings	0.02	0.02	0.0004	0.019	1.94	0.001	0.15
6	Mulch	0.1	0.1	0.01	0.097	9.71	0.037	3.70
7	Soil	0.7	0.36	0.252	0.68	67.96	0.933	93.31
Total		1.03	0.089714286	0.2701	1	100	1	100

COOLER: 2 NASA JSC OFFICE WASTE

Total weight of the sample received= 4.49 kg

Overall Bulk Density= 0.116 kg/L

Average Bulk Density=0.209 KG/L

Table A-2. NASA JSC Office waste composition

S.N.	Components	Wt(kg)	Bulk Density (kg/L)	Vol (L)	Wt Fr	Wt%	Vol Fr	Vol %
1	Paper Bags	0.17	0.10	0.017	0.0417	4.17	0.0069	0.69
2	Cotton	0.22	0.12	0.0264	0.0540	5.40	0.0108	1.08
3	Dirty Clothes	0.02	0.20	0.004	0.0049	0.49	0.0016	0.16
4	Empty Gatorade Bottle	0.05	0.05	0.0025	0.0123	1.23	0.0010	0.10
5	Empty Cans	0.02	0.02	0.0004	0.0049	0.49	0.0002	0.02
6	Carpet	1.7	0.90	1.53	0.4175	41.75	0.6250	62.50
7	Trash Bag (Polythene)	0.005	0.09	0.00045	0.0012	0.12	0.0002	0.02
8	Thermocol	0.03	0.08	0.0024	0.0074	0.74	0.0010	0.10
9	Office Papers	0.02	0.40	0.008	0.0049	0.49	0.0033	0.33
10	Paper Towels	0.02	0.11	0.0022	0.0049	0.49	0.0009	0.09
11	Paper Glasses	0.03	0.03	0.0009	0.0074	0.74	0.0004	0.04
12	Cups	0.02	0.02	0.0004	0.0049	0.49	0.0002	0.02
13	Straw	0.01	0.01	0.0001	0.0025	0.25	0.0000	0.00
14	Plastic Food Container	0.3	0.30	0.09	0.0737	7.37	0.0368	3.68
15	Cardboard	0.6	0.60	0.36	0.1473	14.73	0.1471	14.71
16	Food Box	0.2	0.20	0.04	0.0491	4.91	0.0163	1.63
17	Cardboard Food Box	0.6	0.60	0.36	0.1473	14.73	0.1471	14.71
18	Cigarette Box	0.05	0.05	0.0025	0.0123	1.23	0.0010	0.10
19	Tissue Paper	0.007	0.10	0.0007	0.0017	0.17	0.0003	0.03
	TOTAL	4.072		2.44795	1	100	1	100

COOLER: 3 NASA JSC CAFETERIA WASTE

Total weight of the sample received= 2.90 kg

Overall Bulk Density= 0.112 kg/L

Average Bulk Density=0.160 kg/L

Table A-3. NASA JSC Cafeteria waste composition

S.N.	Components	Wt (kg)	Bulk Density (kg/L)	Vol (L)	Wt fr	Wt %	Vol fr	Vol %
1	Cardboards	0.57	0.60	0.342	0.2027	20.27	0.746	74.64
2	Big Plastic Bag	0.19	0.09	0.017	0.067	6.75	0.03	3.73
3	Plastic Food Containers	0.21	0.03	0.006	0.0746	7.46	0.014	1.375
4	Uneated/Adhered Food	1.83	0.05	0.091	0.650	65.0	0.2	19.9
5	Tissue Paper	0.005	0.10	0.000	0.001	0.17	0.00	0.10
6	Paper Towels	0.007	0.11	0.000	0.0024	0.24	0.00	0.16
Total		2.81		0.458	1	100	1	100

Table A-4. NASA JSC Cumulative Methane Yield Results

Type of Waste	Dry Weight (g)	VS (g)	Total Methane (L)	Cumulative Methane Yield (L CH ₄ /g VS) thermophilic	Cumulative Methane Yield (L CH ₄ /g VS) @ STP
Office Papers Run1	98.41	57.49	22.12	0.3848	0.32
Office Papers Run2	98.41	57.49	16.35	0.28	0.24
Cardboard Run1	59.90	55.00	16.15	0.2936	0.24
Cardboard Run2	98.04	90.02	15.14	0.17	0.14
Paper Towels Run1	98.10	89.07	16.84	0.19	0.16
NASA JSC Low Mix Combination	98.50	65.80	7.48	0.11	0.09
NASA JSC Landscaping Waste	4.70	4.124	0.87	0.17	0.14

APPENDIX B
PILOT SCALE STUDY: OPERATION OF A SEMI-CONTINUOUS ANAEROBIC
DIGESTER UNDER THERMOPHILIC CONDITIONS

The digester set-up is shown in Figure B-1. A stainless steel conical bottom digester was constructed with a total volume of 45 gallons. The height and inner diameter of the cylindrical section were 0.74 m (29 inches) and 0.4 m (16 inches), respectively. The height and diameter of conical section were 0.558 m (22 inches) and 0.4 m (16 inches), respectively. Conical section was modified to achieve easy separation of digested solids from bottom of the digester. Modified conical section is as shown in Figure B-1.

Cylindrical and conical sections were bolted together by using a gasket. A heating jacket was constructed by winding a copper coil around the cylindrical section. Hot water from an electrical heater was recirculated through the copper coil. Complete digester was insulated using kjsdhfkhsdak foam. Ports for recirculation were constructed at different heights on cylindrical and conical sections. For the present study, recirculation of the leachate was done as shown in Fig.B- 1 which was the only mode of mixing in the digester.

Solids were fed to the digesters by operating the knife gate valves at the top. Top gate valve (# 8) was opened first and the chamber was filled with the solids. Then, this valve was closed and valve 7 was opened which allowed solids to fall in the digester; thus making sure no air got into the system. Digested solids were withdrawn from the bottom in a similar way.

A gas port was provided at the top of digester. A gas sampling port with rubber septa was made in the gas line from the digester to the U-tube gas meter. Gas production from the digesters was measured using a positive displacement gas meter.

The device consisted of a clear PVC U-tube filled with anti-freeze solution, a float switch (Grainger), a counter (Redington Inc.) and a solenoid valve (Fabco Air). The U-tube gas meter was calibrated in-line to determine volume of biogas per count. A count was considered as that amount of gas read on syringe (in milliliters) for which the gas meter completes one whole number count (e.g. one count = 0.25 L, then two counts = 0.5 L and continued on). The pH in the digester was measured daily using pH meter (Accumet pH meter, Model 805 MP).

The feedstock was provided by Tropicana, company based in Tampa. It consisted of citrus seeds, peels and pulp after juicing operation. The feedstock was frozen at -20°C and fed directly to the digester whenever needed without bringing it to the room temperature. No pretreatment was given to the feedstock. Different characteristics of the feed are provided later.

Average nutrient composition of dried citrus pulp is given in Table B-1. Analyses were obtained by the Feed Laboratory, Division of Chemistry, Florida Department of Agriculture, Tallahassee. All mineral values are expressed on a dry matter basis.

Initially, the anaerobic digester was run on sugar beet tailings from American Crystal Sugar. About 1 kg of solid feed was added once a week for a month. After consistency in biogas production, citrus waste was fed to the digester at a consistent feed rate. Initially, 1 kg of wet citrus waste was fed to get an idea of total biogas which could be produced by complete digestion.

It is reported that anaerobic digestion of citrus pulp is not a common practice because of the toxic effect of peel oils on anaerobic bacteria. If the waste stream is

sufficiently stripped off d-limonene, an anaerobic digester can reduce the BOD by 90% with methane content in the biogas ranging from 60- 70 %.

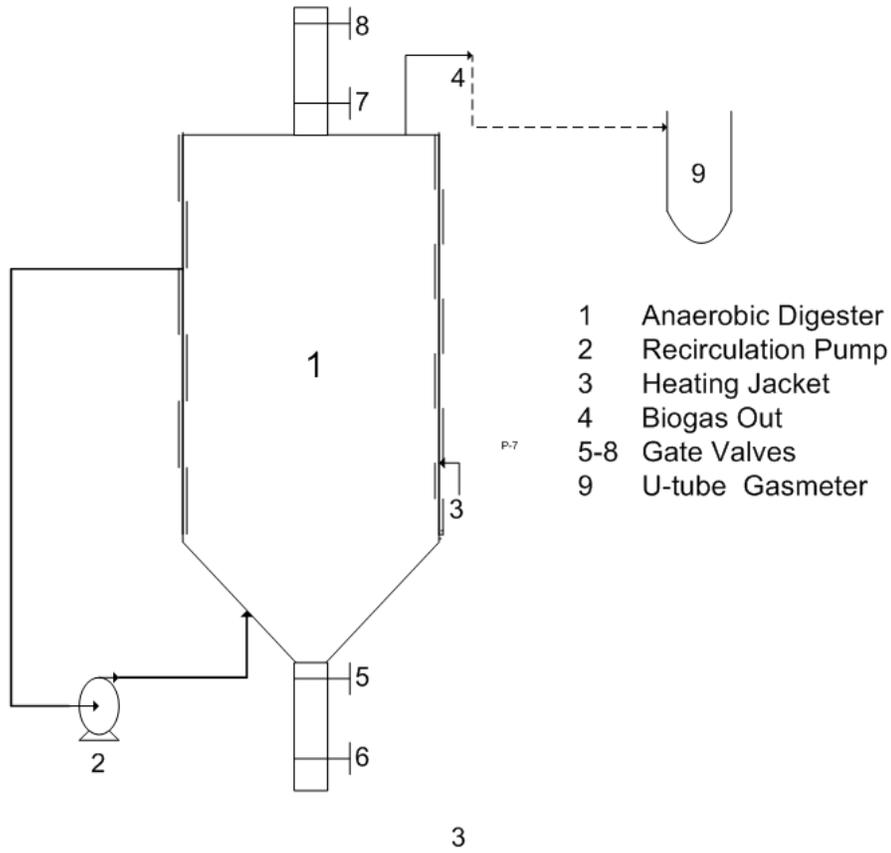


Figure B-1. Digester set up for pilot scale studies

Table B-1. Feed analysis of Citrus waste

Nutrient	Content
Moisture, %	8.58
Ash, %	4.68
Ether extract, %	3.74
Crude protein, %	6.16
Crude fiber, %	12.28
N.F.E., %	64.56
Calcium, %	1.43
Phosphorus, %	0.11
Magnesium, %	0.12
Potassium, %	1.09
Sodium, %	0.096
Sulfur, %	0.066
Iron, ppm	98.72
Copper, ppm	6.19
Zinc, ppm	9.94
Manganese, ppm	5.7
Cobalt, ppm	0.073

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BIOGRAPHICAL SKETCH

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