A Study of Energy Recovery Potential from Domestic Sewage Sludge

by

Abi Aiman Bin Ayoub

A dissertation submitted in partial fulfillment of
the requirements of the
Bachelor of Engineering (Hons)
(Mechanical Engineering)

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Universiti Teknologi PETRONAS
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CERTIFICATION OF APPROVAL

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Approved by,

(Ir. Dr. M. Shiraz Aris)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
May 2011

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

ABI AIMAN BIN AYOUB

ABSTRACT

Domestic sewage sludge can be defined as a soft mud and a slimy precipitate product produced from sewage. The objectives for this project are to determine the energy recovery potential of domestic sewage sludge and to calculate the amount of energy that can be recovered from daily sludge disposal. This project focuses on the characterization of secondary sewage sludge and the amount of energy recovery based on Feed-in-Tariff (FiT). Secondary sludge was chosen as it contains lesser moisture content compared with primary sludge. The problem with sewage sludge is mainly cost. The cost for disposal is too expensive. It also causes the cost of running sewage treatment plant to increase. There is also very limited landfill for sludge disposal. In this work, the sewage sludge was characterized by Thermal Gravimetric Analysis (TGA) for proximate analysis to determine the moisture content, volatile matter, fixed carbon and ash content, and CHNS analyzer for ultimate analysis to determine the C, H, N and S composition in the secondary sewage sludge. While the heating value of the sewage sludge was determined by using Bomb Calorimeter. A few experiments for energy profiling and energy content with time dependency were carried out. The heating value obtained from the experiment was 15.54 ± 0.2 kJ/ g. It was found that, the heating value of secondary sewage sludge is time dependent: the energy content was reduced more than 1 kJ/g after 7 days. Based on this finding, secondary sewage sludge is not recommended to be used for energy recovery after 7 days. Based on the proximate analysis by using Thermal Gravimetric Analyzer (TGA), moisture content in the secondary sludge was 10%, volatile matter 49.5%, Ash content 28.6% and Fixed Carbon 21.9%. The ultimate analysis using CHNS analyzer gave the result of Carbon 34.06%, Hydrogen 4.45%, Nitrogen 6.28% and Sulfur 1.1%. Based from this study, it was found that the amount of sludge to be disposed in landfill can be reduced by using the sludge to generate electricity; consequently health problem can be prevented. The power generated from this energy recovery study can be used in reducing the cost of running sewage treatment plant as well.

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CHAPTER 1

INTRODUCTION

1.1 Background of study

Energy recovery from sewage sludge becomes very important since it can reduce environmental impact from disposing sewage sludge and minimize the amount of sewage sludge to be disposed in landfill. Therefore, a study of energy recovery potential from domestic sewage sludge becomes a good option for green technology and reducing the cost of running sewage treatment plant.

In general, sludge can be referred to as dirt, usually associated with pollution, contamination and disease. It is considered universally as a waste, something to be disposed from out of sight and out of mind. Sludge can be defined as a soft mud or mire, a slimy precipitate produced from sewage. As far as an environmental engineer is concerned, sludge is a by-product from several sources [1, 2].

These sources are as follows:

- Water treatment plant
- Sewage treatment plants
- Dredging of rivers and harbours
- Coal and sand washeries
- Industrial manufacturing
- Agriculture

In this research, secondary sludge was used since the moisture content of secondary sludge is lower compared to primary sludge. When the moisture content is high, more energy will be used to dry the sludge before the conversion of energy from heat to electrical energy.

The first step in this study is the characterization of secondary sewage sludge. The characterization and energy profiling will be involved with combustion properties and chemical composition. Some of the parameters involved will be moisture content, heating value and chemical properties which can be determined from proximate and ultimate analysis.

In general, the sludge may contain 80-98% moisture. There are many processes involved before sludge can be disposed. A flow chart for sludge handling is shown in Figure 1.1 [3]:

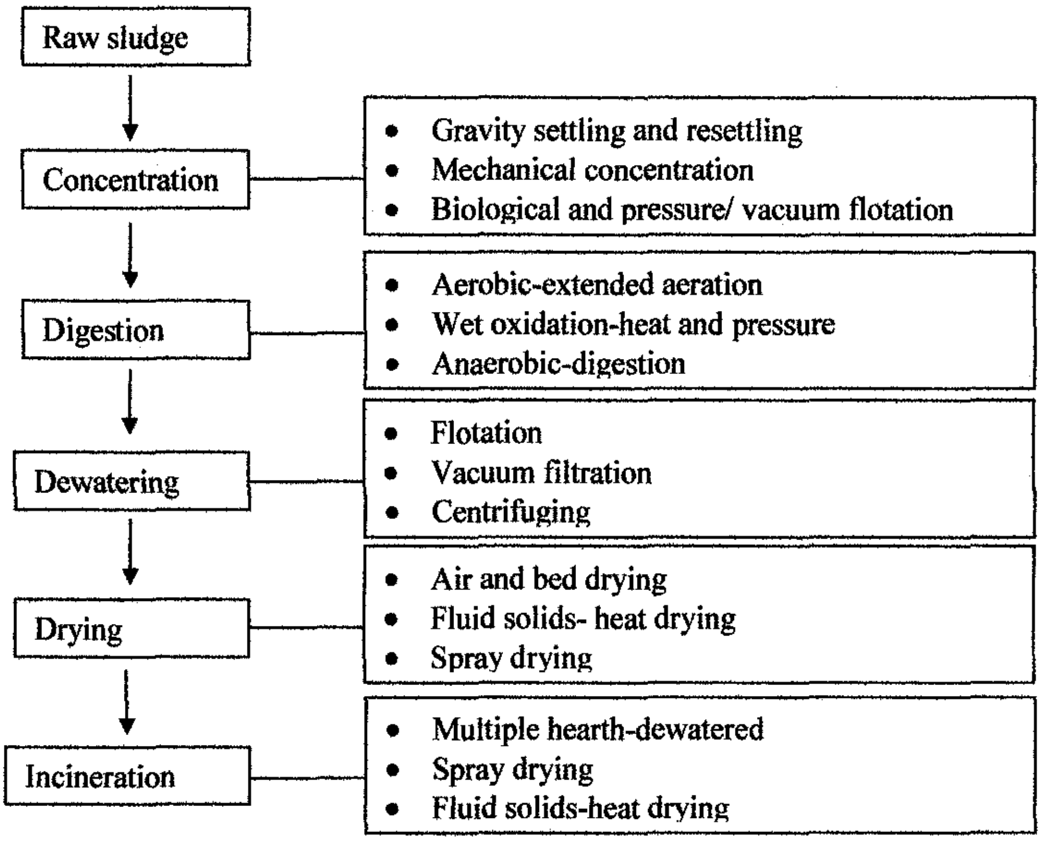

Figure 1.1: Flow chart for sludge handling

An understanding of sludge handling is helpful for determining the suitable process for experimental process and power generation strategy in energy recovery plant. Based on the literature review, there are variations in the results for heating value and chemical composition. The variations may come from the treatment process, location and also age of the sludge after the dewatering process. Therefore, it is a necessary to characterize on Malaysia domestic sewage sludge as a function of time before considering its potential for energy recovery.

1.2 Problem Statement

Domestic sewage sludge contains organic matters, nutrients and minerals suspended in a water medium. These waste products are potentially valuable resources for energy recovery. The cost to dispose domestic sewage sludge is high which is about RM 140 400 per month. As the result, the cost for running sewage plant will also be high. Limited landfill for sludge disposal has been an issue concerning sludge disposal. Sewage sludge also contains pathogenic bacteria, viruses and protozoa along with other parasitic helminthes, which can give rise to potential hazards to the health of humans, animals and plants, if it is not dispose properly [4]. Hence, energy recovery from domestic sewage sludge will reduce the area needed for landfill and reduce the environmental impact on sludge disposal. Therefore, energy recovery of these resources becomes a good option.

1.3 Objective

The objectives of this research are:

- To determine the energy content in sludge over a period of time in order to get the best sludge condition for energy recovery plant.
- To determine the mass fuel burning rate of the sewage sludge for energy recovery purposes to generate a minimum power output of 2 MW.
- To calculate the amount of energy that can be recovered from daily production of sewage sludge.

1.4 Scope of Study

This research has been conducted mainly using secondary sewage sludge as the moisture content for secondary sewage sludge is lower compared to that of primary sludge. Due to its low moisture content, secondary sewage sludge is considered better than primary sludge for energy recovery purposes since a lot of energy can be saved from drying process.

In this research, 2 MW steam turbine was used to produce electricity and the mass fuel burning rate for daily sludge disposal amount of 60 000kg need to be calculated to determine the amount of energy that can be recovered.

CHAPTER 2

LITERATURE REVIEW

2.1 Energy Policies

According to the Tenth Malaysia Plan (10th MP) for the year 2011-2015, the Prime Minister, Datuk Seri Najib Tun Razak has announced Malaysia's voluntary commitment to reduce up to 40% of carbon emissions intensity relative to Gross Domestic Product (GDP) by 2020. The government has formulated a National Renewable Energy Policy and Action Plan [5]. There are five policies in this action plan which as stated below:

1) To increase Renewable Energy (RE) contribution in the national power generation mix.
2) To facilitate the growth of the RE industry
3) To ensure reasonable RE generation costs
4) To conserve the environment for future generation
5) To enhance awareness on the role and importance of RE

To achieve rapid development of renewable energy (RE), the government is turning to Feed-in Tariff (FiT), a mechanism that allows electricity produced from RE resources like solar, biomass, biogas, mini-hydro, solid waste and wind to be sold to power utilities at a fixed premium price for a specific duration. Malaysia's FiT mechanism is widely expected to be modeled after Germany has introduced the FiT system [6].

FiT is a policy to encourage renewable energy development that include guaranteed purchase of electricity generated from renewable sources at price higher than the calculated price of production of electricity. The policy was pioneered in Denmark and Germany and has spread to 17 other European nations. FiT is widely recognized as the most successful policy in the world for encouraging renewable energy development [7].

Malaysia's FiT mostly prioritizes biomass and small hydroelectric generation, with incentives for the first 9MW of installed Photovoltaic (PV) in 2011, increasing every year to 165MW in 2015. Malaysia currently gets less than 1% of its electricity from renewable energy sources. The Ministry of Energy predicts that the FiT would increase this amount to 5.5% by 2015 [7]. Therefore, it is proven that, our government is very serious to go into green technology.

According to Greentech Malaysia, the FiT will enable anybody, whether individual consumers or companies, to generate renewable electricity and sell it to the utilities at a premium price for a fixed period of time, usually 20 years [8].

Therefore, by conducting a study about energy recovery potential from domestic sewage sludge, it will at least contribute to increase the percentage of renewable energy and support our government's emphasis on renewable energy.

2.2 Sewage Sludge

According to a technical report for Connecticut Resources Recovery Authority (CRRA) [9], there are 3 forms of sludge that can be utilized for energy recovery:

1) Liquid sludge (solids 3-6%, generated in settling tanks and basin).
2) Sludge cake (mechanical dewatering of liquid sludge, dry solid is 15-30%).
3) Dried sludge, solid greater than 90%, produced by thermal drying.

In this research, the focus is on dried sludge or dewatered sludge. Currently there are 3 common methods to dewater sludge [10]:

1) Drying beds

   This is the oldest and the most common method for dewatering sludge. This method involves shallow rectangular beds which are divided into small compartments. The beds have porous bottoms made of materials such as ash. Sludge is poured over the porous bed up to a height of 125 to 250 mm. Drainage of water occurs through the bed, and finally collected and removed at the bottom.

   In addition to dewatering by drainage, evaporation occurs at the surface under the action of wind and sun. Cracking of dried sludge provides further surface for evaporation. Dried sludge is removed either manually or mechanically. Deep drying beds can also be used but they have the disadvantage of prolonged drying period.

2) Pressure filtration

This is a type of batch process which has metal chambers lined with filter material like filter cloth. The metal back of chambers has grooves. Conditioned sludge is pumped into the chambers at a pressure of about 600-850 kPa.

At this pressure solids are retained adjacent to the cloth while the liquid drains through the grooves and drawn out at the bottom. Compressed sludge further acts as filter for incoming sludge

3) Vacuum filtration

This method is a continuous process. There is a revolving segmented drum, partially submerged in a shallow tank which contains conditioned sludge. When a vacuum of about 90 kPa is applied to the submerged segments, sludge is sucked onto the surface of the filter cloth.

As the drum rotates, the layer of sludge on the surface of filter comes out of the tank. Dewatering occurs and finally the sludge is removed by a scraper blade. The cycle is repeated again and again.

These 3 methods are very costly and research has been done to improve the methods. According to [11], there are 3 main methods of thermal sewage-sludge disposal, which are:

1) Combustion: Multiple hearth furnace, fluidized bed, smelting and rotary furnaces
2) Co-combustion: With coal, other fuel or with Municipal Solid Waste (MSW)
3) Alternative process: Pyrolysis, oil from sludge, gasification and hybrid methods

2.3 Sludge treatment and disposal

Sludge treatment depends on the amount of solids generated and other site-specific conditions. Composting is most often applied to small-scale plants with aerobic digestion for midsized operations, and anaerobic digestion for the larger-scale operations [12].

Composting works by mixing the sludge with a bulking agent to make sure that the mixture can be aerated for an accelerated aerobic degradation process. The problems are more energy is required to aerate the composting material, the end product of the compost must be disposed at a suitable land, and it is not an attractive choice to farmers due to a low fertilizer value [12].

Aerobic digestion is the process of oxidizing and decomposing the organic part of the sludge by micro-organisms in the presence of oxygen. Aerobic digestion produces a stable product, reduces mass and volume, and reduces pathogenic organisms. It also has some key advantages for smaller plants when compared to anaerobic digestion such as low capital equipment cost and simple operational control [13].

Anaerobic digestion works by feeding the sludge to an enclosed reaction tank where naturally occurring bacteria degrade the organic material. The end products are biogas and stabilized sludge. The biogas can be converted to both electricity and heat. There are no actual problems related to anaerobic digestion other than the problem of disposing the digested sludge. The normal outlet is as a fertilizer to land. However, not enough suitable land is available and sludge can be incinerated [14]. In Malaysia, dewatered sludge is disposed at designated landfill.

2.4 Sewage Sludge Combustion

In order to determine the potential energy recovery, several parameters must be considered for suitability of sewage sludge as a fuel or energy source: for example moisture content, heating value and the chemical properties obtained from proximate and ultimate analyses [15].

The heating value of sewage sludge for treatment plants in Canada is shown in table 2.1[16]. Samples were taken from various stages in a treatment plant.

Table 2.1: Measured Parameters for Raw Wastewater and Sewage Sludge Samples [16]

Sample	**Heating value (kJ/g dry)**
Raw waste water	3.2±0.1
Primary sludge	15.9±0.2
Secondary sludge	12.4±0.1
Anaerobically digested sludge	12.7±0.3

A mathematical model, based on chemical composition, to predict the heating value of domestic wastewater sludge (DWS) in Thailand was proposed by [17]. The data used in this study were collected from 20 different waste water treatment plants around Thailand. The samples were dried under sunlight for 1 to 2 days prior to their characterization. The results confirmed the composition of sewage sludge to be mainly made of volatile matter and ash ranging from 42.4% - 53.2% and 60.2% - 80.3% of the total sample composition respectively. The amount of fixed carbon was measured to be approximately 11.8% of the sample's chemical composition and the heating values ranged from 4000 kJ/g to 14,000 kJ/g. For the ultimate analysis, the value of fixed carbon ranged between 9% and 31%, Hydrogen between 2% and 4.2%, Nitrogen between 1.5% and 4.3%, Sulfur between 0.4% and 2% and Oxygen between 15.7% and 24.3%.

According to Jianguo Jiang, Xuejuan Du and Shihui Yang (2010), a study in the combustion characteristics of domestic wastewater sludge (DWS) using Thermal gravimetric method for samples collected in China, the dry basis heating value was found to be 10.8 kJ/g, approximately half the published value of low grade coal. The content or fixed carbon in the total composition of the sludge samples was found to be 3.48%, a relatively low value for fuels, while the content of volatile matter stood at 43.67%, a quantity which can help to promote efficient combustion.

Table 2.2: Heating value from various sources of biomass [19, 20, 21]

Fuel Name	Heating Value (MJ/kg)
Coal	23-28
Almond shells	19.8
Almonds hulls	20.0
Oil Palm Fiber	19.06
Oil Palm Shell	17.78
Bagasse	13-17
Saw Dust	20.93
Olive pits	22.0
Olive husk	21.8
Rice Husk	15.61
Rice straw	18.7
Poultry sludge	27-30
Switchgrass	19.9
Municipal Solid Waste (MSW)	21.03

Based on the literature review, there are variations in the results for heating value and chemical composition. The variations may come from the treatment process, location dependant and also age of the sludge after the dewatering process. Therefore, it is a necessary to characterize on Malaysia domestic sewage sludge as a function of time before considering its potential for energy recovery.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

The research has been conducted according to the methodology outlined in the flowchart shown in figure 3.1.

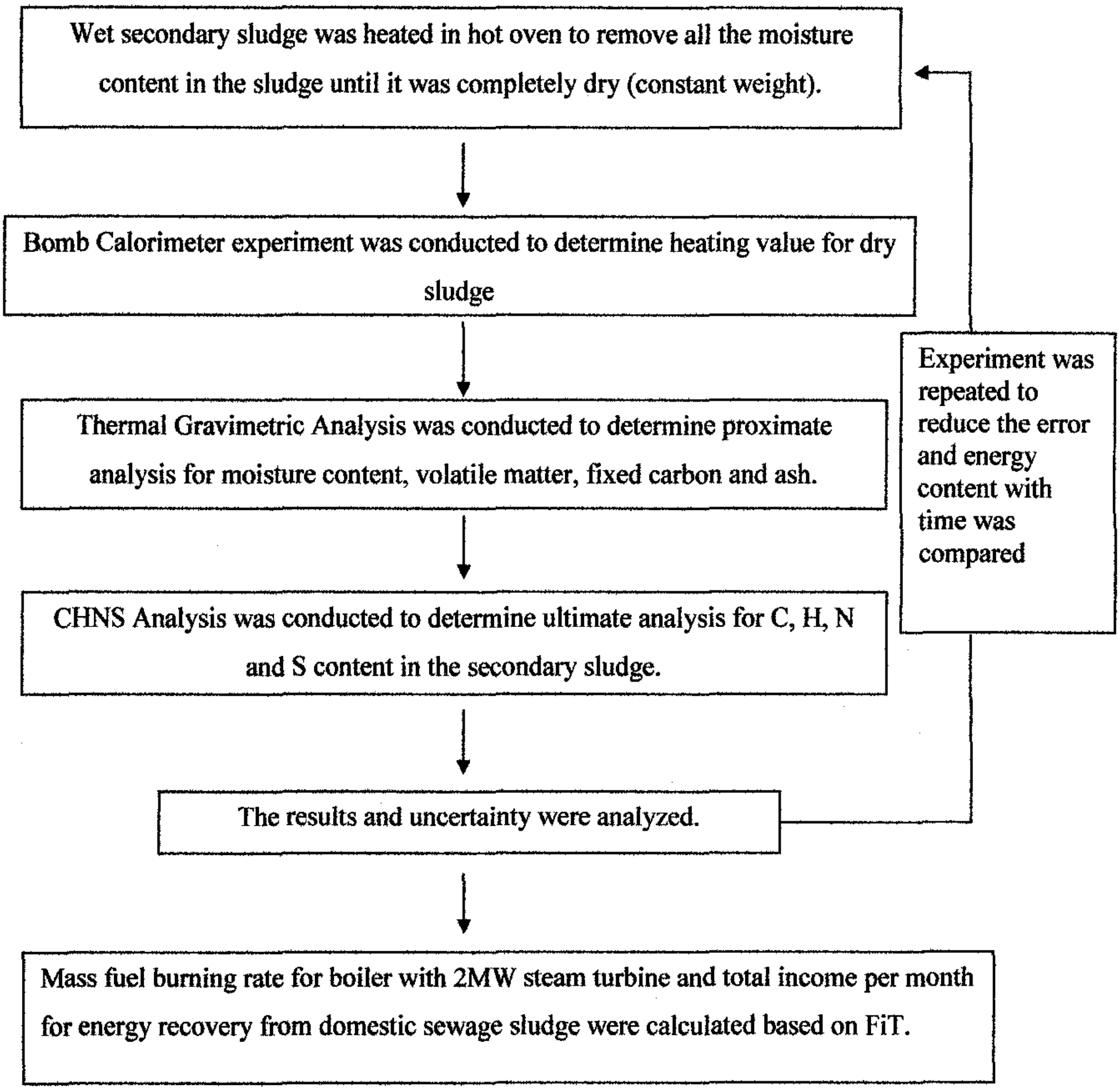

Figure 3.1: Research methodology

3.2 Gantt Chart for FYP I

NO	ACTIVITIES	WEEK NO/MONTH													
	A STUDY OF ENERGY RECOVERY POTENTIAL FROM DOMESTIC SEWAGE SLUDGE	**JULY**		**AUGUST**				**SEPTEMBER**				**OCTOBER**			
		1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
1	**Selection of Project Topic**							**Mid Semester break**							
	1.1 Writing proposal	■													
	1.2 Submission of proposal		■												
2	**Preliminary Research Work**														
	2.1 Study about sludge combustion		■	■											
	2.2 Study about sludge characteristic			■											
	2.3 Conduct experiment with bomb calorimeter			■											
3	**Submission of preliminary report**														
	3.1 Writing preliminary report				■										
4	**Project Work**														
	4.1 Conduct experiment to determine energy profile in sludge					■									
	4.2 Analyze experiment result					■									
	4.3 Continue with energy recovery study					■	■								
5	**Submission of Progress Report**								■						

6	**Seminar**								■						
7	**Project Work**														
	7.1 Repeatability of energy profiling experiment		■	■	■	■	■		■	■	■	■	■	■	
8	**Submission of Interim Final Draft**														■
9	**Oral Presentation**														■

Figure 3.2: Gantt chart for FYP I

3.3 Gantt Chart for FYP II

NO	**ACTIVITIES**	**WEEK NO/MONTH**													
	A STUDY OF ENERGY RECOVERY POTENTIAL FROM DOMESTIC SEWAGE SLUDGE	**JAN**		**FEB**				**MARCH**				**AUGUST**			
		1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
1	**Uncertainty Analysis**							Mid Semester break							
	1.1 Uncertainty analysis for experimental result	■	■	■	■										
	1.2 Submission of uncertainty analysis	■	■	■	■										
2	**Expansion of Experiment Database**														
	2.1 Bomb Calorimeter		■	■	■	■	■								
	2.2 CHNS		■	■	■	■	■								
	2.3 TGA														

3	**Submission of Progress Report**														
	3.1 Writing progress report					■	■		■						
4	**Project Work**														
	4.1 Calculation on stoichiometry equation and actual calculation				■	■	■		■	■	■	■	■	■	■
	4.2 Calculation on power generation output				■	■	■		■	■	■	■	■	■	■
	4.3 Finalize reports and results											■	■	■	■

Figure 3.3: Gantt chart for FYP II

3.4 Experimental

The samples for the experiment were taken from secondary sewage sludge waste water treatment plant in Kuala Lumpur. To remove the moisture, the samples were dried in an oven at 105°C. During the heating, the samples were intermittently weighed until they reached a constant weight to ensure that the samples have been completely dried. The parameters for characterizing the energy profile for domestic sewage sludge were gathered from the bomb calorimeter, proximate analysis and ultimate analysis. The heating value procedure for bomb calorimeter was carried out in accordance to ASTM D2015. The proximate analysis involved the use of Thermal gravimetric Analyzer (TGA-Perkin Elmer PYRIS1TGA) to determine moisture content (MC), volatile matter (VM), fixed carbon (FC) and ash content. The samples for the TGA analysis were weighed at 10 mg each, and analyzed in the TGA with a heating rate of 20 °C/min from 100°C to 850°C under an air flow of 20 mL/min. The ultimate analysis involved the use of CHNS analyzer (Leco CHNS-932, VTF-900) to determine the Carbon (C), Hydrogen (H), Nitrogen (N) and Sulfur (S) content in the sample.

3.4.1 Removal of Moisture Content in Secondary Sewage Sludge

Before any experiment can be done on secondary sewage sludge, it must be dried to remove the moisture content. The detailed procedure can be referred to in APPENDIX 1-1. The sludge was dried in 5 different containers to obtain 5 samples.

Figure 3.4: (Left) The oven used for drying, and (right) the dried secondary sewage sludge

After a few hours, the weight became constant, which indicated that all the moisture content had been totally removed during this process.

Next, the sample was kept in a plastic bag to protect the sample from moisture in the air. It was very important to ensure that the samples were free from moisture before any experiment can be done on the samples.

Figure 3.5: Secondary sewage sludge samples after drying

3.4.2 Heating Value Analysis by using Bomb Calorimeter

The ASTM D2015 procedure for determining heating value for domestic sewage sludge was used. The detailed procedure can be referred to in APPENDIX 1-2. In order to reduce error, each sample was analyzed twice, and the average reading from the two experimental run is reported. The precision error for bomb calorimeter obtained from the experiment is ±0.1 kJ/g

Figure 3.6: Bomb Calorimeter

Before measuring the heating value for secondary sewage sludge, a standard sample was used to determine the variance between actual results and theoretical results. In this case, benzoic acid was used.

Table 3.1: Heating value for standard sample

Sample	**Heating Value for Standard Sample (Benzoic Acid) (kJ/g)**	**Measured Heating Value for Standard Sample (kJ/g)**	**Difference**	**Error percentage (%)**
1	26.46	26.42	0.04	(0.04/26.46)x100=0.15
2	26.46	26.45	0.01	(0.01/26.46)x100 = 0.04
	Difference Average (kJ/g)		**0.025**	**(0.025/26.46)x100 =0.1**

Therefore, the calibration error for the equipment is ±0.1 kJ/g

3.4.3 Proximate Analysis Using Thermal Gravimetric Analyzer

In this experiment, the parameters determined are moisture content (MC), volatile matter (VM), fixed carbon (FC) and ash content. The detailed procedure can be referred to in APPENDIX 1-3. Samples of 10 mg each were analyzed in the TGA with a heating rate 20° C/min from 100°C to 850°C under an air flow of 20 mL/min. This equipment has a precision error of 0.001%.

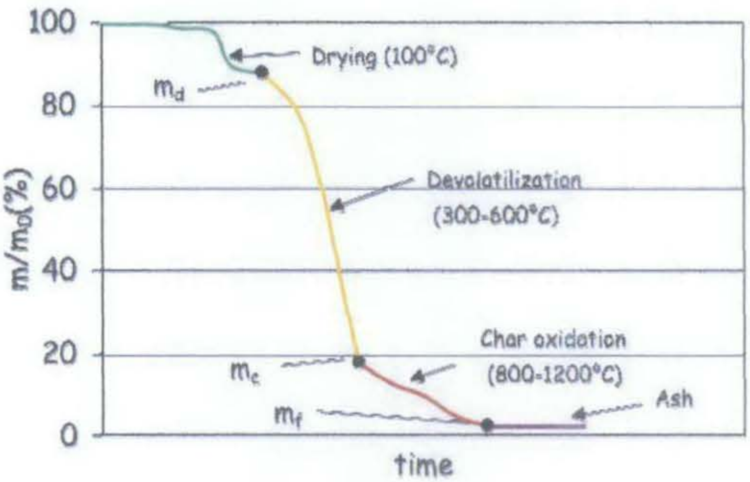

Figure 3.7: TGA Analysis

Figure 3.7 shows the sample weight decreases as the temperature rises in the oven (TGA). The sample started to lose its volatile components (devolatilisation or pyrolysis) at around 300-600°C. The solid residue after devolatilisation called char and contained mainly carbon (fixed carbon) and ash. The char oxidized at 800-1200°C in presence of oxygen [22].

The data for TGA are usually presented on dry basis [22]. Each parameter determined by TGA is calculated according to the following formulas:

1) Moisture content (%) = 100 $-m_d$
2) Volatile matter (%, moisture free (mf)*)=$(m_d-m_c)/m_d$ x 100
3) Ash content (%,mf)=m_f/m_d x100
4) Fixed carbon (%,mf)=100-volatile matter (%.mf)-ash content (%,mf)

*Often expresseses as dry

3.4.4 Ultimate Analysis using CHNS Analyzer

The ultimate analysis involved the use of CHNS analyzer (Leco CHNS-932, VTF-900) to find the elemental composition for Carbon (C), Hydrogen (H), Nitrogen (N) and Sulfur (S) content in the sample. The detailed procedure is provided in APPENDIX 1-4. The samples were prepared in the small tin capsule with sample weight ranging from 1.5-2 mg each. A standard sample was prepared to find the variance between actual and theoretical value. The standard sample used in the experiments was Sulfamethazime.

Table 3.2: CHNS Standard sample composition using Sulfamethazime

Sample	C (%)	H (%)	N (%)	S (%)
1	53.34	5.665	22.21	12.38
2	52.67	5.059	23.51	12.41
3	51.78	5.07	20.13	11.52
4	54.88	4.09	21.16	13.15
5	53.77	4.53	20.65	10.53
Average	53.29	4.88	21.53	12.00
Actual value	51.78	5.07	20.13	11.52
Error difference (%)	(1.51/51.78)x100 = 2.92%	(0.19/5.07)x100 = 3.74%	(1.4/20.13)x100 = 6.95%	(0.48/11.52)x100 = 4.17 %

The error difference is still within acceptable values since it does not exceed 10%. Therefore the results from this experiment or analysis are still reliable.

The result from this ultimate analysis is important for calculating boiler efficiency and in determining stoichiometry equation for the combustion of secondary sewage sludge as fuel.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Proximate Analysis

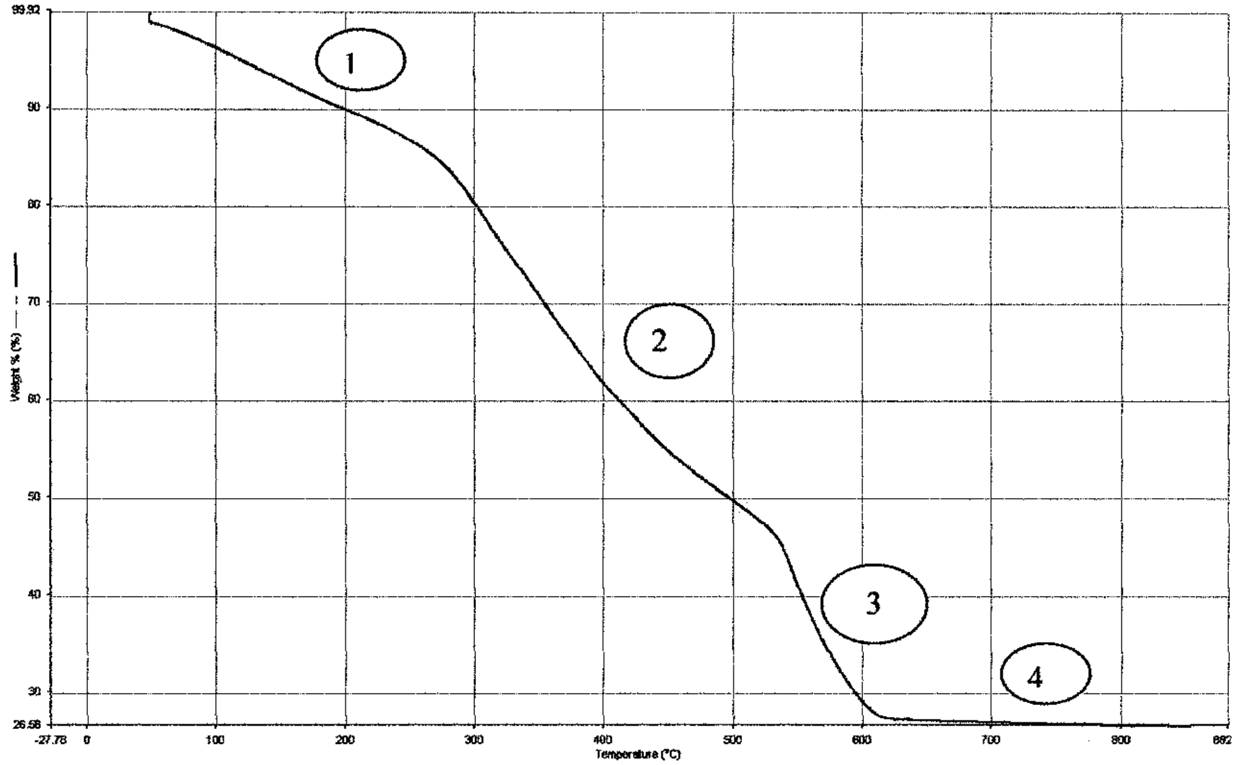

Figure 4.1: Thermal Gravimetric Analyzer (TGA) Result

The TGA result (figure 4.1), shows that the sample weight decreases as the temperature increases. The first slope represents moisture content, the second slope indicates for volatile matter content, the third slope indicates fixed carbon and the last part of the graph marked as region 4 in Figure 4.1 indicates the ash content. The data usually presented on dry basis. Example calculation for each parameter is given below:

Moisture content at 200°C = 100-90 = 10%

Volatile matter at 520°C (%, moisture free, mf), V = (99-47)/99 x 100 = 52.5%

Ash content at 880°C (%, mf) = (26.58/99) x 100 = 26.8%

Fixed carbon (%, mf), F = 100-52.5-26.8= 20.7%

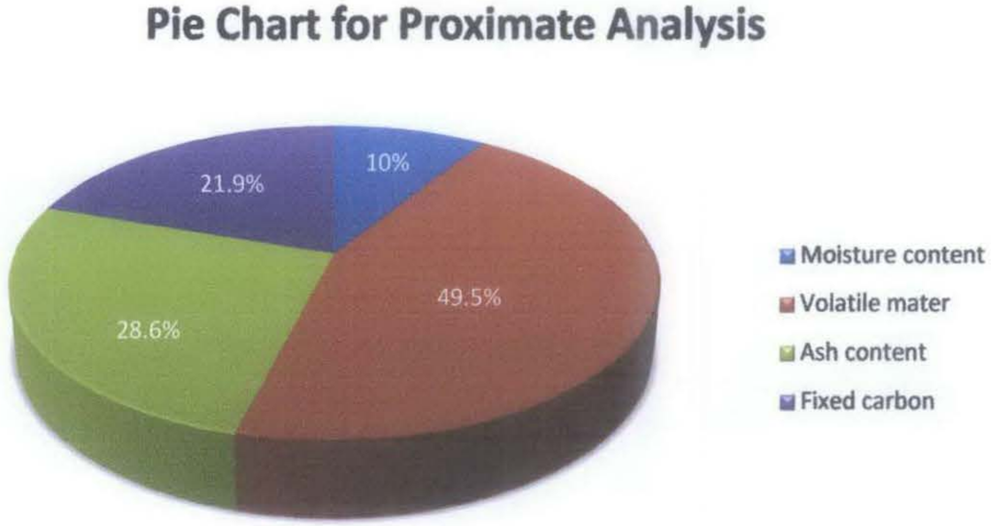

Figure 4.2: Pie Chart for Proximate Analysis

For the TGA experiment, 15 tests were conducted and the average value was taken. The average value is stated in Figure 4.2. According to the previous work done by Puchong Thipkhunthoda (2005), the value for volatile matter is still within the range which is between 42.4% and 53.2%. On the other hand, the value for the ash content is lower than the reported value is about 28.6%. The amount of the fixed carbon in this study is higher than the value previously reported by the Puchong Thipkhunthoda. This is due to the variation in the moisture content between the previous work and present work.

The lesser moisture content in the sludge is better since high moisture content and ash content in secondary sewage sludge can cause ignition and combustion problem. Thus it will reduce the combustion efficiency.

4.2 Ultimate Analysis

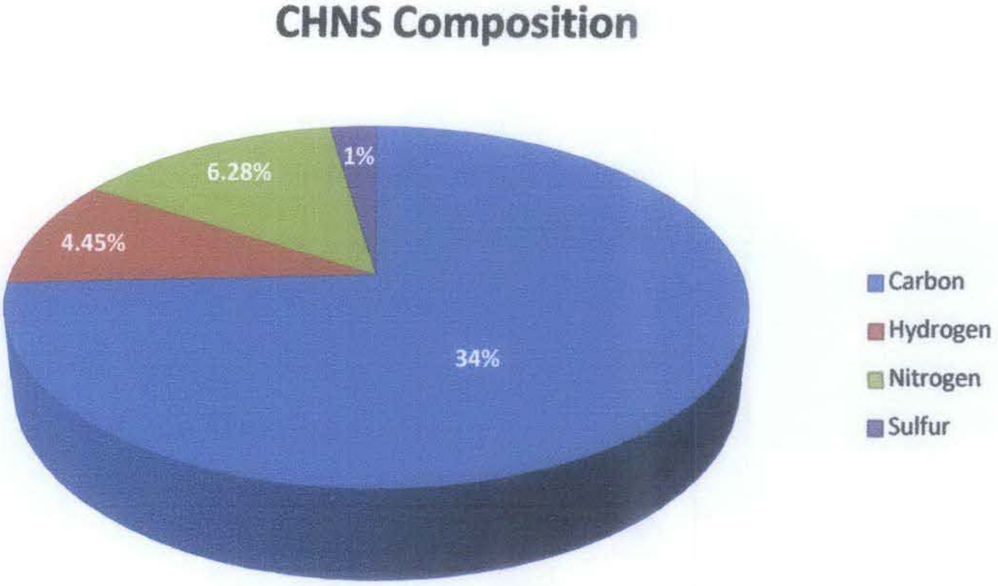

Figure 4.3: Pie Chart for Ultimate Analysis

Based on Figure 4.3, the sample was found to contain 34.06% of carbon, Hydrogen 4.45%, Nitrogen 6.28% and Sulfur 1.10%. This chart is generated based from the average value of 15 tests.

According to the previous work done by Puchong Thipkhunthoda (2005), the ultimate analysis of sewage sludge for values of Carbon range between 9 and 31%, Hydrogen between 4.2 to 20%, Nitrogen between 1.5 and 4.3% and sulfur between 0.4 and 2%.

Based on this comparison, this CHNS result is acceptable as the values are still within the range. The value of available Nitrogen is relatively high compared with the previous work because the calibration/ instrumentation error for nitrogen is quite high which is about 6.95% compared to the calibration/ instrumentation error for C, H and S during the test for standard sample. This result is important for developing stoichiometry equation and calculation for boiler efficiency.

4.3 Initial Moisture Content

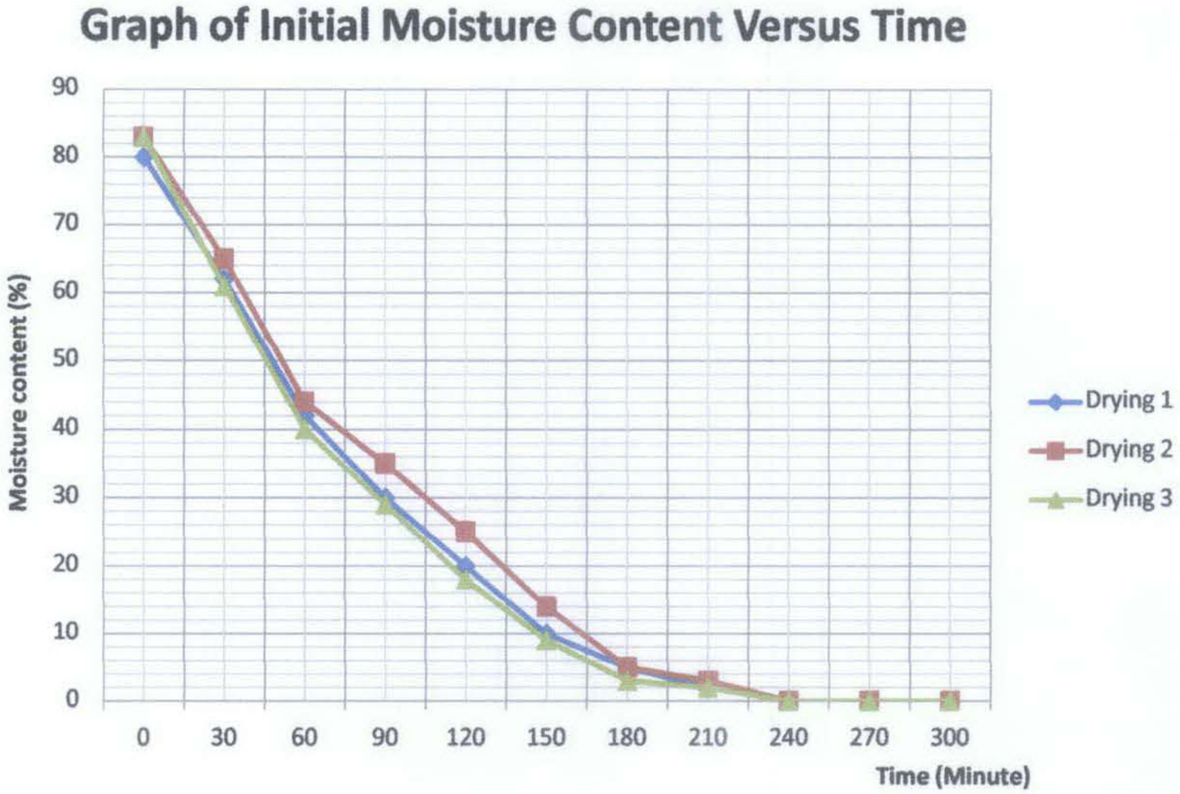

Figure 4.4: Initial moisture content value

Referring to Figure 4.4, the initial moisture content for domestic secondary sewage sludge in Malaysia is ranges from 80-90%. The secondary sewage sludge is completely dry when the weight measured during the drying process in the oven is constant. The time taken during the experiment to completely dry the sludge was around 5 hours.

This initial moisture content value is very important for calculating the energy needed for the drying process. Lower moisture content is the better as lesser energy would be required for drying.

4.4 Uncertainty Analysis for Heating Value (Bomb Calorimeter)

As the best sludge handling for energy recovery is during the first 7 days after dewatering process, this research has been focused on the heating value for this period of time. The mean heating value for this period is 15.54 kJ/ g with the highest uncertainty value of ± 0.19 kJ/g

Table 4.1: Heating value for different time period

	Heating Value (kJ/g)									
Sample	1	2	3	4	5	6	7	8	9	10
1 month old sample	14.25	14.42	14.46	14.25	14.44	14.52	14.45	14.44	14.45	14.44
After 1 week old sample	14.68	14.44	14.39	14.53	14.42	14.43	14.42	14.41	14.33	14.05
Before 1 week old sample	15.37	15.59	15.49	15.43	15.64	15.7	15.8	15.7	15.0	15.7

Precision error for bomb calorimeter = ± 0.1

Calibration error using standard sample = ± 0.1

Number of sample, n = 10

Standard deviation, s = $\sqrt{\frac{1}{N}\sum_{1=1}^{N}(x-\bar{x})^2}$

$$\bar{X}=\frac{(15.37+15.59+15.49+15.43+15.64+15.7+15.8+15.7+15+15.7)}{10}$$

$\bar{X} = 15.54$ kJ/g

$\sum_{i=1}^{N}((x-\bar{x})^2$=$(15.37-15.54)^2+(15.59-15.54)^2+(15.49-15.54)^2+(15.43-15.54)^2+(15.64-15.54)^2+(15.7-15.54)^2+(15.8-15.54)^2+(15.7-15.54)^2+(15.0-15.54)^2+(15.7-15.54)^2$

$\sum_{i=1}^{N}((x-\bar{x})^2 = 0.4920$

s = $\sqrt{\frac{1}{10}\sum_{i=1}^{10}(\,(x-\bar{x})^2}$

s= $\sqrt{\frac{1}{10}\sum_{i=1}^{10}(0.4920)}$

s= 0.2218

A 95% confidence level

α= 1-0.095= 0.05

From table t-distribution

For v=n-1

v=9

$\alpha/2$= 0.025

$t_{\alpha/2}$ = 2.262

$\mu = \bar{x} \pm t_{\alpha/2} \frac{s}{\sqrt{n}}$

$\mu = 15.54 \pm 2.262(0.2/\sqrt{10})$

μ= 15.54 ± 0.14

Bias error = 0.14

Uncertainty,RSS=$\sqrt{(\text{Precision error})^2 + (\text{Bias error})^2 + (\text{Calibration error})^2}$

RSS = $\sqrt{0.1^2 + 0.14^2 + 0.1^2}$

RSS = ±0.2 kJ/g

Heating value = 15.54 ±0.2 kJ/g

Table 4.2: Uncertainty Distribution for Bomb Calorimeter Results

Uncertainty, RSS (kJ/g)	**Sample**									
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
1 month	0.15	0.14	0.14	0.15	0.14	0.14	0.14	0.14	0.14	0.14
1 week	0.15	0.14	0.14	0.14	0.14	0.14	0.14	0.14	0.14	0.16
1 day	0.15	0.14	0.14	0.14	0.14	0.15	0.15	0.15	0.19	0.15

4.5 Comparison of Heating Value with time

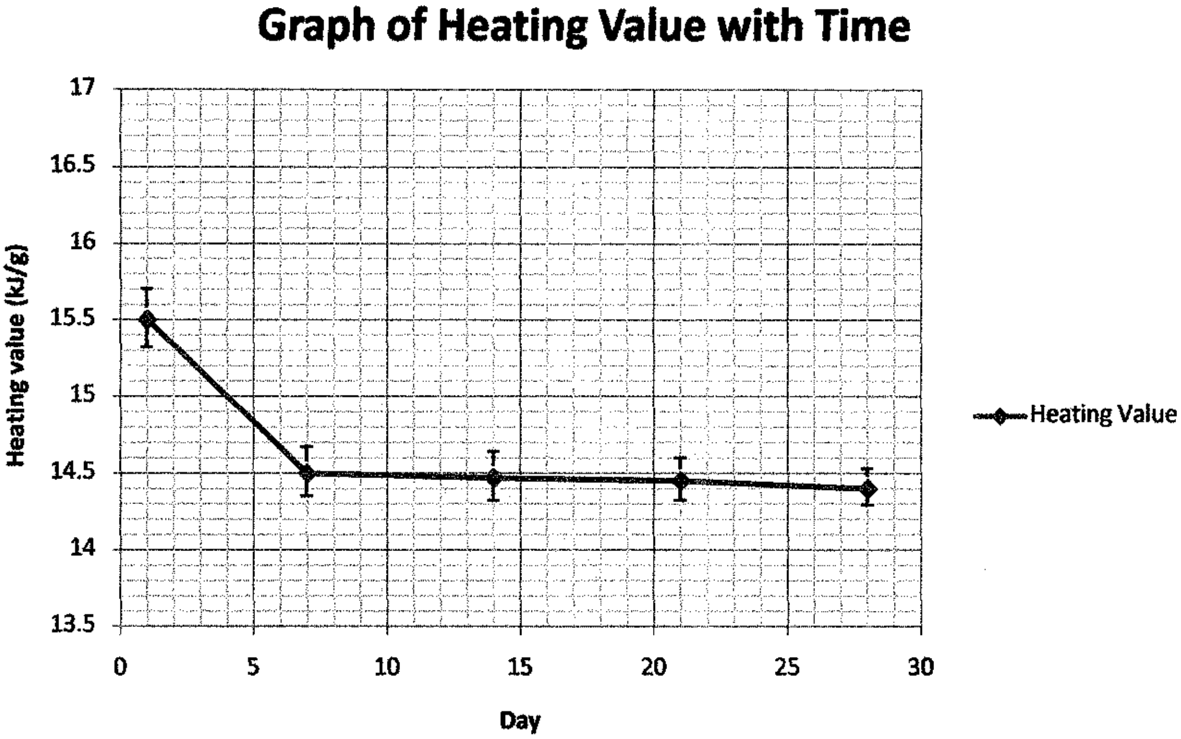

Figure 4.5: Relationship between heating value with time

Referring to the error bar in Figure 4.5, the uncertainty value for one day old sample does not overlap with the uncertainty value for seven days old sample. Therefore, the heating value measured during the experiment was firm with low uncertainty value.

Based on Figure 4.5, it can be concluded, the energy content in the secondary sewage sludge is dependent on time. The most significant change in energy content for secondary sludge is between 1 day old and 1 week old sample. After 1 week, there is not much variation in the energy content of the sludge.

The variation in energy content with time is due to changes in the carbon content in the sample over time. The highest carbon content for below 7 days old sample was 35.53% and the highest carbon content for sample one month old sample was 32.16%. Therefore, it is best to perform energy recovery from dried secondary sewage sludge within 7 days after drying.

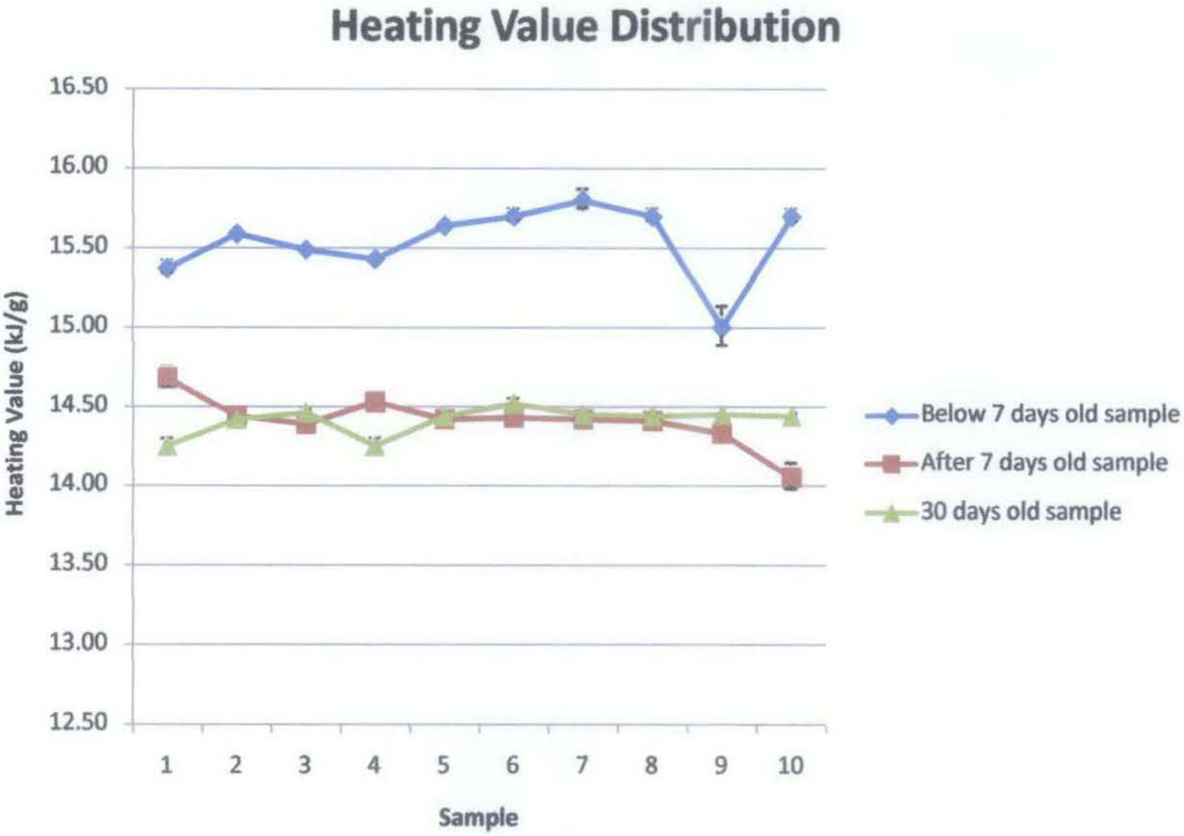

Figure 4.6: Heating value distribution with error bar

By referring to Figure 4.6, the highest heating value is 15.7 kJ/g with the highest uncertainty value of ±0.19 kJ/ g. The heating value is higher for the sample less than 7 days old and starts to decrease by 1 kJ/g after 7 days. The variation in heating value after 7 days until 1 month is about 0.02 kJ/g, which is considered as not significant.

Based on this result, the best time for energy recovery is before 7 days since the energy content in the secondary sewage sludge is maximum.

4.6 Prediction of Heating Value

$HHV = 255.75V + 283.88F - 2386.38$ .... (1)

% Variation = 100 x ($HHV_{predicted}$ - $HHV_{measured}$)/ ($HHV_{measured}$) .... (2)

For comparison between predicted heating value and measured heating value for High Heating Value (HHV), an empirical equation 1 is used and the percentage of variation of the result is calculated by using equation 2 which was developed in [17].

$HHV = 255.75V + 283.88F - 2386.38$

Where,

V = volatile matter content

F = fixed carbon content

For the dried sludge sample used in this work, the volatile matter content is 49.5% and fixed carbon content is 21.9%. Thus the predicted HHV is calculated to be 16.69 kJ/ g and it percentage variation is 5.76%

$HHV = 255.75(49.5) + 283.88(21.9) - 2386.38$

Predicted HHV = 16.49 kJ/g

% Variation = 100 x (16.49 -15.54)/16.49

% Variation = 5.76%

The variation came from the moisture content. Even though the sample was thought to be completely dry as indicated by the constant weight, there was still 10% moisture content in dried sample as shown by the proximate analysis result.

During the drying process the mass of sludge was weighted until it was constant to ensure that all the moisture content had been removed. However there was a possibility that moisture content in the air which affected the result of proximate analysis. For better result, this experiment needs to be done in a dry environment where the moisture content is low in order to get small variations in the predicted and measured heating values. The result was compared with the previous work done another researcher and it was found that the calculated variation is still within the range reported in [7].

4.7 Comparison of heating values between biomass and coal

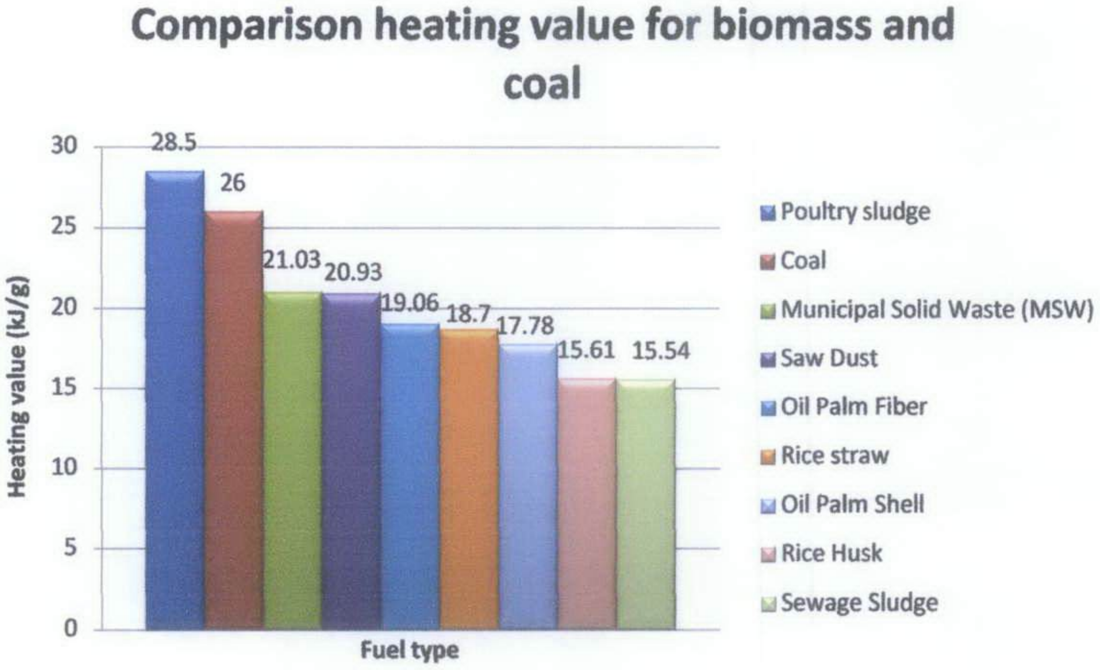

Figure 4.7: Comparison of heating values for biomass

Referring to Figure 4.7, the heating value measured for sewage sludge in this study is not as high as coal and it is close to the heating value for rice husk and oil palm fibre which is between 15-20kJ/g.

In comparison to poultry and coal, the heating value for the sewage sludge is lower, but the main important point of this study is to know the amount of energy that can be potentially recovered from 60 000 kg of sludge, the daily amount of sludge disposal by Indah Water, Kuala Lumpur. This is very important as when the sludge can be utilized for energy recovery, then the problems associated with landfill needed for its disposal and the cost of running sewage treatment plant can be reduced.

4.8 Energy Balance for Power Generation

After the sludge is dried, it can be used as fuel in the boiler to heat water, and produce steam which is fed into the steam turbine. In this study, a steam turbine with 2 MW was used as the minimum target power output to calculate the mass fuel burning rate for the sewage sludge. Boiler efficiency was taken as 0.86 with the inlet temperature 160°C. This was taken from the literature review [23] since for boiler efficiency calculation, proper boiler need to be fabricated to burn the sewage sludge and the product from the sewage sludge combustion need to be analyzed using gas analyzer. This is still in the development process, therefore proper equipment is not available yet. A detailed calculation for boiler efficiency is explained in Chapter 5 in the recommendation section.

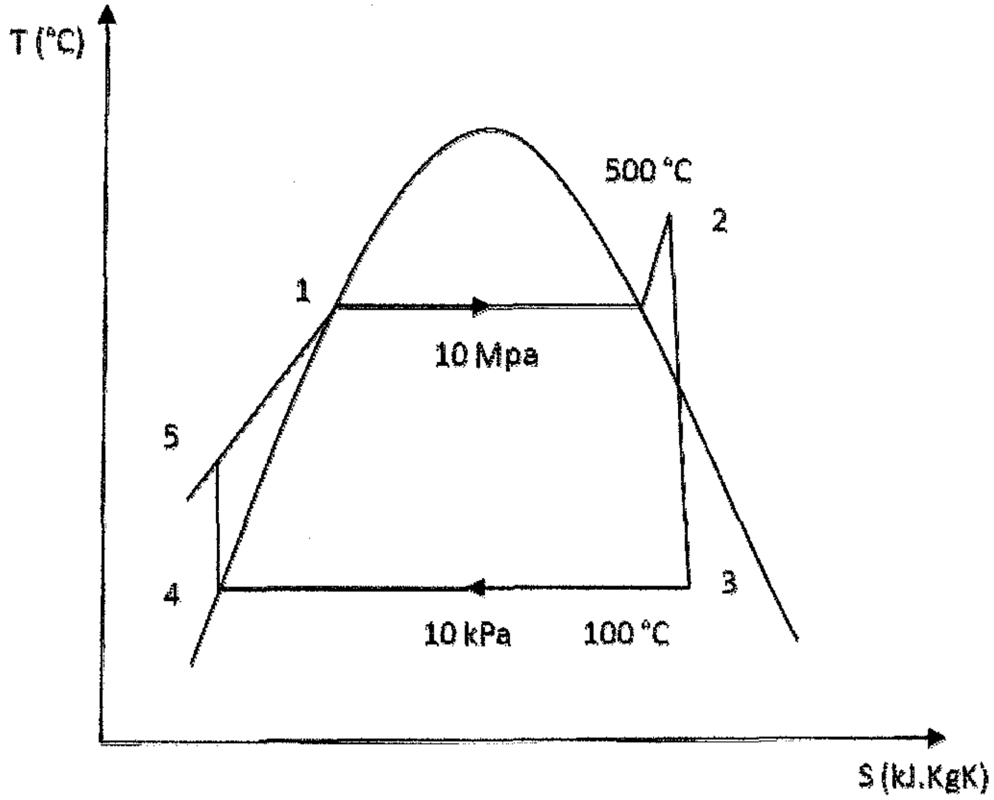

Figure 4.8: T-s diagram for energy recovery plant

Turbine

Mass flow rate for steam, $\dot{m}_s$

$\dot{m}_s\,(h_2 - h_3) =$ Power Output $= 2 \times 10^3$ kW

From superheated steam table at T=500°C, P= 10 MPa

$h_2 = 3374.8$ kJ/kg

h_3 is a mixture

$h_3 = h_f + x_3 h_{fg}$

$s_2 = 6.6011$ kJ/kg.K

$s_2 = s_3$

$s_3 = s_f + x_3 s_{fg}$

$x_3 = (s_3 - s_f)/s_{fg}$

At P =10 kPa

$S_f = 0.6479$ kJ/kg.K

$S_{fg} = 7.5055$ kJ/kg.K

$x_3 = (s_3 - s_f)/s_{fg}$

$x_3 = (6.6011 - 0.6479)/7.5055$

$x_3 = 0.7932$ kJ/kg.K

$h_3 = hf + x_3 h_{fg}$

At P=10 kPa

$h_f = 191.46$ kJ/kg , $h_{fg} = 2393.29$ kJ/kg

$h_3 = 191.46 + 0.7932(2393.29)$

$h_3 = 2089.82$ kJ/kg

$\dot{m}_s\,(h_2 - h_3) =$ Power Output $= 2 \times 10^3$ kW

$$\dot{m}_s = \frac{2 \times 10^3}{3374.8 - 2089.82}$$

$\dot{m}_s = 1.556$ kg/s

Boiler

Fuel burning rate, $\dot{m}_f$

Energy lost by the boiler = Energy gained by the steam

$$\dot{m}_f c_v \eta_{boiler} = \dot{m}_s(\mathrm{h}_2 - \mathrm{h}_5)$$

h_5 is compressed liquid. It can be approximated using saturated water table at T=160°C

h_5= 675.5 kJ/kg

$$\dot{m}_f c_v \eta_{boiler} = \dot{m}_s(\mathrm{h}_2 - \mathrm{h}_5)$$

$$\dot{m}_f = \frac{\dot{m}_s(\mathrm{h}_2 - \mathrm{h}_5)}{c_v \eta_{boiler}}$$

$$\dot{m}_f = \frac{1.556(3374.8 - 675.5)}{(15.54\,x\,10^3)(0.86)}$$

$$\dot{m}_f = 0.3142 \text{ kg/s}$$

$$\dot{m}_f = 0.3142\ \frac{\text{kg}}{\text{s}}\ \text{x}\ \frac{3600\text{ s}}{\text{hr}}$$

$$\dot{m}_f = 1131.12 \text{ kg/hr}$$

Daily amount of sludge disposal is 60 000 kg

About 20% only from the total 60 000 kg of sludge is solid while 80 % is water.

Dried sludge = 0.2(60 000)

Dried sludge = 12 000 kg

Time = (12 000 kg/1131.12kg/hr)

Time = 10.6 hours

Total power generated = 2MW(10.6hr) = 21.2 MW

Therefore, the daily total sludge weight to be disposed which is about 60 000 kg can be used to generate 2 MW of electricity for about 10.6 hours with the mass fuel burning rate of 1131.12 kg/hr.

Power Calculation for Sludge Drying

The secondary sewage sludge consists of 80% liquid and 20% solid. Based on the proximate analysis, even though the sludge was dried in the oven until the mass was constant, but there was still about 10% of moisture which cannot be neglected and it is associated with the dried sludge. Therefore, when calculating the power for sludge drying, this 10% will be considered in the calculation. In actual application, heat loss also needs to be considered.

Latent heat of evaporation (water) = 2257kJ/kg

Specific heat capacity for water= 4.186 kJ/kg.K

Mass of wet sludge = 0.1185 kg

Water, 80%= 0.0948 kg

Dried sludge, 20% = 0.0237 kg

Final moisture content is 10% which is 0.0119 associated with 90% of the sludge which is 0.1067 kg

So, (0.0119 x 0.0237)/ 0.1067 = 0.0026 moisture is associated with the dried sludge.

During the drying process, 0.1185kg of sludge will lose (0.0948-0.0026) moisture = 0.092 kg moisture.

Power for drying process = (heat to raise temperature to 105°C + latent heat to remove water)/ (5hours x 3600 s)

Power for drying = [(105-25)(4.186)(0.1067) + (0.092)(2257)] / (5 x 3600)

Power for drying = 243 kJ/ (5 x 3600)

Power for drying = 0.0135 kW

Power for drying per kg = 0.0135/0.1185 =0.114 kW/kg

Total sludge to be disposed daily by Indah Water Kuala Lumpur is 60 000 kg. 48 000 kg is water that need to be removed.

Power for removal of 48 000 kg of water = 0.114 kW/kg x 48 000 kg

Total power = 5472 kW

Total power = 5.472 MW

Total power generated with steam turbine = 21.2 MW

Nett power output = 21.2-5.472= 15.72 MW

4.9 Feed in Tariff Calculation

Referring to the tariff from Kementerian Tenaga, Teknologi Hijau dan Air (KeTTHA) for installation capacity below 10 MW, the Feed-in-Tariff (FiT) rate for biomass is RM0.31/kWh. Additional RM 0.01 for use of steam based electricity generating systems with overall efficiency of above 14%. Therefore the FiT is RM 0.32 / kWh.

On a daily basis, the total power generated for 60 000 kg of secondary sludge by steam turbine

= 2000 kW x 10.6 hr

= 21200kWh

Total income from power generated

= 21200 kWh x RM 0.32/kWh

= RM 6784

If the plant runs for 30 days/ month, the total income

= RM 6784 x 30

= RM 203 520

The cost for sludge disposal monthly is RM 140 400. Therefore, the energy recovery will contribute in reducing the cost of running sewage sludge especially in sludge disposal operation.

Hence, instead of disposing the sludge, energy recovery is a good option. Thus, environmental impact can be reduced and money is saved.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Based on the conducted research, domestic sewage sludge has the potential for energy recovery use. The objective of this research to determine the energy contained in sludge as a variation with time has been achieved successfully, where the best time for sludge handling for energy recovery is below 7 days. In conclusion, the energy contained in sludge varies with time during the first 7 days after the dewatering process of sludge. After 7 days, the variation in the energy content for dewatered secondary sewage sludge is not very much, which is only about 0.02 kJ/kg compared to the variation within the first 7 days which is about 1.02 kJ/ kg. Therefore, the secondary sewage sludge is suitable to be used in the energy recovery plant if the sludge is not more than 1 week old to prevent more energy loses.

From the analysis of the combustion properties for secondary sewage sludge, the heating value is 15.54 kJ/g with the uncertainty of ±0.2 kJ/g. This high heating value gives a good combustion property for energy recovery. This result is used in the power generation to get mass burning rate for secondary sewage sludge as fuel to produce 2 MW power output from steam turbine. The mass burning rate for secondary sewage sludge is 1131.12 kg/h. Therefore, the total sludge weight to be disposed daily which is about 60 000 kg can be used to generate 2 MW electric for about 10.6 hours period.

This research has been successfully done to identify the possible solution in green technology to reduce the environmental impact concerning sludge disposal. Based from this study, it is found that the amount of sludge to be disposed in landfill can be reduced by using the sludge to generate electricity; consequently health problem can be prevented. The power generated from this energy recovery study can be used in reducing the cost of running sewage treatment plant as well.

5.2 Recommendation

It is recommended to fabricate a boiler and the sludge sample is burned in the boiler, and the product of the combustion is analyzed with a gas analyzer to know the percentage of the product. An example calculation for boiler combustion efficiency is given here for future reference.

For example, during the sludge combustion the product of the combustion is given as CO_2 13.6%, O_2 4.4%, CO 3.4% and SO_2 0.02%. By using the data from ultimate and proximate analysis, here is the calculation for the boiler combustion efficiency

Table 7: Fuel mole fractions

	Mass fraction, mf_{orig} (kg)	mf_i (kg_i/kg_{total})	Molecular weight, (kg_i/kg_{mole})	$(mf/MW)_i$ ($kgmole/kg_{total}$)	$\tilde{x}_i$ ($kgmole/kgmole_{total}$)
C	0.34	0.45	12	0.03728	0.4345
H_20	0.1	0.13	18	0.00731	0.0852
H_2	0.045	0.06	2	0.02961	0.3450
O_2	0.2	0.26	32	0.00822	0.0958
N_2	0.063	0.08	28	0.00296	0.0345
S	0.01	0.01	32	0.00041	0.0048
Ash	0.24	-	$\sum(mf/MW)_i$	0.0858	-

Where, for example, for carbon, C

$mf_i = 0.34/(1-0.24) = 0.45$

$(Mf/MW)_i = 0.03728 + 0.00731 + 0.02961 + 0.00822 + 0.00296 + 0.00041$

$(Mf/MW)_i = 0.0858$ kgmole/kg

$\tilde{x}_i = 0.03728/ 0.0858 = 0.4345$

Stoichiometric equation:

$[0.4345C_s + 0.0852H_2O + 0.3450H_2 + 0.0958O_2 + 0.0345N_2 + 0.0048S]$

$+ a\,[O_2 + 3.76N_2] \longrightarrow bCO_2 + cH_2O + dSO_2 + eN_2$

Carbon atom balance: $b = 0.434$

Hydrogen atom balance: $2(0.0852) + 2(0.345) = 2c$

$c = 0.4302$

Sulfur atom balance: $d = 0.0048$

Oxygen atom balance: $0.0852 + 2(0.0958) + 2a = 0.434(2) + 0.4302 + 0.0048(2)$

$a = 0.5155$

Nitrogen atom balance: $e = 0.5155(3.76) = 1.9383$

Air-fuel ratio:

Molar:

$$\overline{AF} = \frac{(0.5155\,)(4.76)}{(1\text{ kgmole fuel})}$$

$= 2.15$

Mass:

$$AF = \frac{(2.15\text{ kgmoles air})(28.96\frac{\text{kg}}{\text{kgmole air}})}{(1\text{ kgmole fuel})(0.0858\text{ kgmole fuel/kg})^{-1}} = 5.342$$

Actual equation:

$[0.4345C + 0.0852H_2O + 0.3450H_2 + 0.0958O_2 + 0.0345N_2 + 0.0048S]$

$+ a[0.5155]\,[O_2 + 3.76N_2] \longrightarrow b[0.136CO_2 + 0.034CO + 0.0240O_2 + 0.002SO_2 +$

$0.804N_2] + cH_2O$

Carbon: $0.434 = b\,(0.136 + 0.034)$

$b = 2.5529$

Hydrogen: $0.0852 + 0.3450 = c$

$c = 0.4302$

Nitrogen: $0.0345 + a(0.5155)(3.76) = 2.5529(0.804)$

$a = 1.0411$

Boiler Combustion efficiency

Energy Balance:

$Q = \sum_{product} N_i[\overline{h_f} + \Delta\bar{h}]_i - \sum_{reactant} N_j[\overline{h_f} + \Delta\bar{h}]_j$

Refer to Thermochemical Properties Table

$\sum_{product} N_i[\overline{h_f} + \Delta\bar{h}]_i = [(2.5529)(0.136)(-94054 + 958)CO_2 + (2.5529)(0.034)(-26417 + 711)CO + (2.5529)(0.024)(724)O_2 + (2.5529)(0.002)(-20947 + 1016)SO_2 + (2.5529)(0.804)(710)N_2 + 0.4302(-57798 + 825)H_2O$

$\sum_{product} N_i[\overline{h_f} + \Delta\bar{h}]_i = -57660$ cal/gmole

$\sum_{reactant} N_j[\overline{h_f} + \Delta\bar{h}]_j = 0.0852(-68317)H_2O = -5820.6$cal/gmole

Q= -57660 - (- 5820.6) = -51839.4 cal/gmole of fuel

LHV = Q(stoichiometric without $\Delta\bar{h}$)

$LHV = 0.434(-94054)CO_2 + 0.4302(-57798)H_2O + 0.0048(-70947)SO_2$

$- 0.085(-68317)H_2O$

LHV = -60217.7 cal/gmole fuel

η_{boiler} = -51839.4/-60217.7 = 0.86

= 86 %

HHV = 57660 cal/gmole x 4.187 [(kJ/kgmole)/(cal/gmole)] x 0.0858 kgmole/kg

HHV = 20714 kJ/kg fuel ashless (76 kg ashless/100 kg sludge) = 15742.64 kJ/kg sludge

HHV 15.74 kJ/g sludge

In future, the secondary sewage sludge can be fabricated as fuel briquette by mixing with other biomass fuel to get higher heating value. Besides that, there is also a method called torrefaction, where it is a heat treatment process at a certain range of temperature for duration of time which can give low moisture content and increase energy density.

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Appendices

Appendix 1-1

Procedure for Drying the Secondary Sludge in Hot Oven

Machine : Oven

Model : Carbolite 450

Figure A-1: Oven

Standard Operating Procedure

1. Weight of the initial sample is measured
2. Furnace chamber is opened to make sure it is empty.
3. Sample is placed in the furnace chamber
4. Door is closed properly
5. Oven controller is switch on
6. Required temperature is set by using up and down arrow (↑↓) button at the controller.
7. The actual temperature will be slowly increased until it reaches the required temperature.
8. The weight for the sample is measured every 30 minutes to make sure all moisture is removed.

Caution: Wear proper personal protective equipment. Make sure to use glove, jacket, shoes, face shield and proper tools when handling the hot materials and equipment

9. After using the oven, power is switch off and oven is cleaned.

Appendix 1-2

Procedure for determine Heating Value using Bomb Calorimeter

Machine : Bomb Calorimeter
Model : C 5000

Figure A-2: Bomb Calorimeter

Standard Operating Procedure

1. Oxygen gas regulator is turned on.
2. Bomb calorimeter and refrigerator batch switch is turn on. 20 minutes is waited for the system stable.
3. Sample is prepared with weigh below 1 gram
4. Sample is placed into crucible. A cotton thread is secured with a loop in on the middle of the ignition wire and placed into decomposition vessel.
5. Sample dialog window is opened to enter the parameters
6. The decomposition vessel is suspended into the filling head of the measurement cell cover

7. Start button is activated. The measurement cell cover is closed. The decomposition vessel is then filed with oxygen. Next, the inner vessel is filled with water. As soon as the system begins with the experiment, a graph of the changes over the time in vessel is showed at display
8. When the measurement is completed, the decomposition is vessel is removed, cleaned and prepared for next experiment.
9. When all the experiment is finished and bomb calorimeter is cleaned. System menu is opened and exit dialog box is opened. Bomb calorimeter, refrigerator bath and regulator oxygen is turned off.

Appendix 1-3

Procedure for Proximate Analysis in Thermal Gravimetric Analyzer

Machine : Differential Scanning Calorimeter

Model : Perkin Elmer Pyris 1 DSC

Figure A-3: Thermal Gravimetric Analyzer

Standard Operating procedure

Start up procedure:

1. Power supply is switch one
2. Computer and intracooler system is turn on
3. Gas regulator for DSC machine is turn on
4. The machine is let running until the sample temperature stabilize at 25°C(Approximately 20 mins)
5. The Pyris 1 DSC software is run.

Sample Preparation:

1. The sample is cut into smaller pieces
2. The empty aluminum sample pan is weight and then the balance is re-zero.
3. The sample is inserted into aluminum sample pan around 5-10mg
4. Sample pan is transferred into sample preparation tool holder. The sample plan I gently clamped.

Running the test sample:

1. The sample is transferred into DSC holder in left side, the right side is reserved for reference sample.
2. At the Pyris 1 DSC program "Method Editor" windows is select and the required parameters is filled.
3. The initial temperature is set and Go to temperature is pressed and the intial temperature is wait to be reached.
4. "Start/Stop" is press to run the analysis.

Shutdown procedure:

1. The intracooler is switch off and the machine is let idle overnight

After operation: Power switch, air and water supply is switch off and equipment is cleaned.

Appendix 1-4

Procedure for Ultimate Analysis in CHNS analyzer

Figure A-4: CHNS Analyzer 932

Standard Operating Procedure

To start the instrumentation of CHNS

1. Helium, Oxygen and compressed air are set to 40 psi
2. Ambient monitor is checked for proper values
3. CO_2, Hydrogen and Sulfur IR cells are monitored to be between 7.5- 9.2 Volts.
4. Oxi. furnace temperature is set o 1000°C
5. Red. Furnace temperature is set to 650°C.
6. Leak check is run if necessary(The gas must be in the 'Analyze' position before a Leak Check can be perfomed).
7. 'Auto/ Manual' switch is set to auto when using carousel or to manual to load samples individually.
8. The gas switch is set to 'Analyze position'.
9. Furnace temperature is waited until is stable.
10. Five blank analyses and 5 standard samples is run.

To shutdown the instrumentation

1. Standby mode is set
2. Oxi. Furnace standby temperature is make sure in 650°C
3. All the gas are switched off
4. Oxi. Furnace temperature is waited to drop to 650°C then the instrument is switched off.

Appendix 2-1

PERCENTAGE POINTS OF THE T DISTRIBUTION

Tail Probabilities									
One Tail		0.10	0.05	0.025	0.01	0.005	0.001	0.0005	
Two Tails		0.20	0.10	0.05	0.02	0.01	0.002	0.001	
D	1	3.078	6.314	12.71	31.82	63.66	318.3	637	1
E	2	1.886	2.920	4.303	6.965	9.925	22.330	31.6	2
G	3	1.638	2.353	3.182	4.541	5.841	10.210	12.92	3
R	4	1.533	2.132	2.776	3.747	4.604	7.173	8.610	4
E	5	1.476	2.015	2.571	3.365	4.032	5.893	6.869	5
E	6	1.440	1.943	2.447	3.143	3.707	5.208	5.959	6
S	7	1.415	1.895	2.365	2.998	3.499	4.785	5.408	7
	8	1.397	1.860	2.306	2.896	3.355	4.501	5.041	8
O	9	1.383	1.833	2.262	2.821	3.250	4.297	4.781	9
F	10	1.372	1.812	2.228	2.764	3.169	4.144	4.587	10
	11	1.363	1.796	2.201	2.718	3.106	4.025	4.437	11
F	12	1.356	1.782	2.179	2.681	3.055	3.930	4.318	12
R	13	1.350	1.771	2.160	2.650	3.012	3.852	4.221	13
E	14	1.345	1.761	2.145	2.624	2.977	3.787	4.140	14
E	15	1.341	1.753	2.131	2.602	2.947	3.733	4.073	15
D	16	1.337	1.746	2.120	2.583	2.921	3.686	4.015	16
O	17	1.333	1.740	2.110	2.567	2.898	3.646	3.965	17
M	18	1.330	1.734	2.101	2.552	2.878	3.610	3.922	18
	19	1.328	1.729	2.093	2.539	2.861	3.579	3.883	19
	20	1.325	1.725	2.086	2.528	2.845	3.552	3.850	20
	21	1.323	1.721	2.080	2.518	2.831	3.527	3.819	21
	22	1.321	1.717	2.074	2.508	2.819	3.505	3.792	22
	23	1.319	1.714	2.069	2.500	2.807	3.485	3.768	23
	24	1.318	1.711	2.064	2.492	2.797	3.467	3.745	24
	25	1.316	1.708	2.060	2.485	2.787	3.450	3.725	25
	26	1.315	1.706	2.056	2.479	2.779	3.435	3.707	26
	27	1.314	1.703	2.052	2.473	2.771	3.421	3.690	27
	28	1.313	1.701	2.048	2.467	2.763	3.408	3.674	28
	29	1.311	1.699	2.045	2.462	2.756	3.396	3.659	29
	30	1.310	1.697	2.042	2.457	2.750	3.385	3.646	30
	32	1.309	1.694	2.037	2.449	2.738	3.365	3.622	32
	34	1.307	1.691	2.032	2.441	2.728	3.348	3.601	34
	36	1.306	1.688	2.028	2.434	2.719	3.333	3.582	36
	38	1.304	1.686	2.024	2.429	2.712	3.319	3.566	38
	40	1.303	1.684	2.021	2.423	2.704	3.307	3.551	40
	42	1.302	1.682	2.018	2.418	2.698	3.296	3.538	42
	44	1.301	1.680	2.015	2.414	2.692	3.286	3.526	44
	46	1.300	1.679	2.013	2.410	2.687	3.277	3.515	46
	48	1.299	1.677	2.011	2.407	2.682	3.269	3.505	48
	50	1.299	1.676	2.009	2.403	2.678	3.261	3.496	50
	55	1.297	1.673	2.004	2.396	2.668	3.245	3.476	55
	60	1.296	1.671	2.000	2.390	2.660	3.232	3.460	60
	65	1.295	1.669	1.997	2.385	2.654	3.220	3.447	65
	70	1.294	1.667	1.994	2.381	2.648	3.211	3.435	70
	80	1.292	1.664	1.990	2.374	2.639	3.195	3.416	80
	100	1.290	1.660	1.984	2.364	2.626	3.174	3.390	100
	150	1.287	1.655	1.976	2.351	2.609	3.145	3.357	150
	200	1.286	1.653	1.972	2.345	2.601	3.131	3.340	200
Two Tails		0.20	0.10	0.05	0.02	0.01	0.002	0.001	
One Tail		0.10	0.05	0.025	0.01	0.005	0.001	0.0005	
Tail Probabilities									

This table was calculated by APL programs written by William Knight. The format of the table is adapted from a table constructed by Drake Bradley, Department of Psychology, Bates College, Lewiston-Ashburn, Maine, U.S.A.