

**CHARACTERIZATION OF WASTE FROM THE PETROCHEMICAL
INDUSTRY**

By

CHE MOHD HASHRUL BIN CHE BAHARUM

Dissertation submitted in partial fulfilment of
The requirements for the
Bachelor of Engineering (Hons)
(Mechanical Engineering)

MAY 2011

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CERTIFICATION OF APPROVAL

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Approved by,



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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.



CHE MOHD HASHRUL BIN CHE BAHARUM

ABSTRACT

Characterization of waste is a study of defining the un- available or undefined data of the waste. The results of this characterization provide a description on the behaviour of waste. Applications are important in many areas of industry, involving metals, plastics, many other materials. In petrochemical industry, characterization of the waste is important to manage their waste output efficiently to reduce cost and time consumptions. This project is about study and research to characterize the behaviour of the waste obtained from the petrochemical industry. The project was done by using the machine and experiments that available in Universiti Teknologi PETRONAS lab. Some of the waste available in the petrochemical industry might be harmful to human and some give benefits. It is important to further the reading on the petrochemical basically the history of the waste itself to learn the characteristic of the waste. The characterization on petrochemical waste is important to assess the potential whether or not the waste can be re-processing for other usage or dumped away. The characterization of this petrochemical waste is not yet being studied for certain waste to overcome the problem on finding it as useful materials. The author's hoping that the results on this research are able give some input to explain about the waste, overcome the problem in order for the waste to be efficiently stored or being reprocessed.

ACKNOWLEDGEMENT

I would like to take this opportunity to express my utmost gratitude to the individuals who have taken the time and effort to assist me in completing this project. Without the cooperation of these individuals, I would undoubtedly have faced complications throughout the course.

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Petrochemical industry is one of the largest industries in this world and it contributes large amount of waste. Some of the waste produce from the petrochemical industry is useful to be reused for example like catalyst waste as a cement substitution but some is being dumped away. Millions of dollars were spent to manage these wastes. Thorough researches have been done to know whether the waste can be used for other usage as useful applications and materials. However, problems occurred during the process like overheat, inappropriate pump ability and even death due to toxic suffocating. The behaviour of waste from petrochemical industry is important to overcome the problem occurred to review the characteristic of the waste as a useful materials.

1.2 PROBLEM STATEMENT

The study of waste immobilization is increasing due to the need to treat and transform waste from the industry into useful. The amounts of waste were increasing by year and affect us in many ways such as pollution and health effect. It is necessary to find alternatives ways of managing the waste. In the petrochemical industry, the waste produced is kept in a designated storage. The purpose of storage is to find out whether it can still be useful or just be dumped away. A large amount of money is being invested to maintain and manage the waste. Various attempts have been made to study and improve this problem. However, the mechanical properties such as rheological behaviour of this waste are not yet being studied in an attempt to explain

this lack of problem. This study is intended to characterize the behaviour of the waste obtained from the petrochemical industry in order to find the benefits of the waste. Efficient storage and reprocess the waste requires good understanding for example rheological behaviour as it relates to waste displacement and pumping requirement. The author's will be using experiment method to identify the wastes characteristics.

1.3 OBJECTIVES AND SCOPE OF STUDY

The objectives of this project are:

- 1 Characterize the undefined behaviour of the waste from petrochemical industry.

The author's project is basically a research of waste from petrochemical industry. Several stages are involved in achieving the objectives of the project. The research work will involve the basic study of rheology, measurements and experimentation. The sample wastes will be obtained from PETRONAS OPU, Malaysia Liquefied Natural Gas Sdn. Bhd and measurement and experiments will be conducted in UTP to understand the behaviour of the waste material at different conditions. This project will be done according to time frame and planned scheduled.

CHAPTER 2

LITERATURE REVIEW

2.1 RHEOLOGY

Rheology is a terminology (Barnes et. al., 1989), used since 1929 when the American society of Rheology was founded. It was conceived by Professor Eugene Bingham of Lafayette College, Indiana. It studies on deformation and flow of matter. The field of study extends the classical discipline of elasticity, described by Hooke's law for solid and Newtonian fluid mechanics, described by Newton's law for fluids, to the flow of material that behave between these classical extremes namely material categorized as being non Newtonian.

Rheological research involves activities in and draws on knowledge from biophysics, chemical engineering, chemistry, computer science, electronics, engineering mechanics, materials science, mathematics, mechanical engineering, medicine and physics among others. Rheology's interdisciplinary nature stems from the variety of materials investigated and the many new questions that must be answered. These are:

1. Rheometry
2. Constitutive equations
3. Measurement of flow behaviour in complex geometries
4. Calculation of behaviour in complex flow.

Every fluid can be studied by a rheological point of view especially when it is necessary to understand its behaviour in certain mechanical conditions. Rheological studies in cement and food industry are two examples of further application of rheology to different fluid used in industrial application. At last a contribution to rheology is given also by energy sector especially concerning the waste from petrochemical industry. My research is dealing with rheology on Petrochemical waste. I will be using the rheometer in the UTP lab

2.2 NEWTONIAN LAW OF PHYSICS

Since the author's project is concentrating on non-Newtonian behaviour matter, it is important to understand what Newtonian behaviour is, in the context of shear viscosity. According to Chabra R.P. Richardson J.F. Non-Newtonian Flow in the Process Industries (2008), Newtonian behaviour in experiments conducted at constant temperature and pressure has the following characteristic:

1. The only stress generated in simple shear flow is the shear stress, the two normal stress differences being zero.
2. The shear viscosity does not vary with shear rate
3. The viscosity is constant with respect to the time shearing and the stress in the liquid falls to zero immediately the shearing is stop. In any subsequent shearing, however long period of resting between measurements, the viscosity is as previously measured.
4. The viscosities measured in different types of deformation are always in simple proportion to one another.

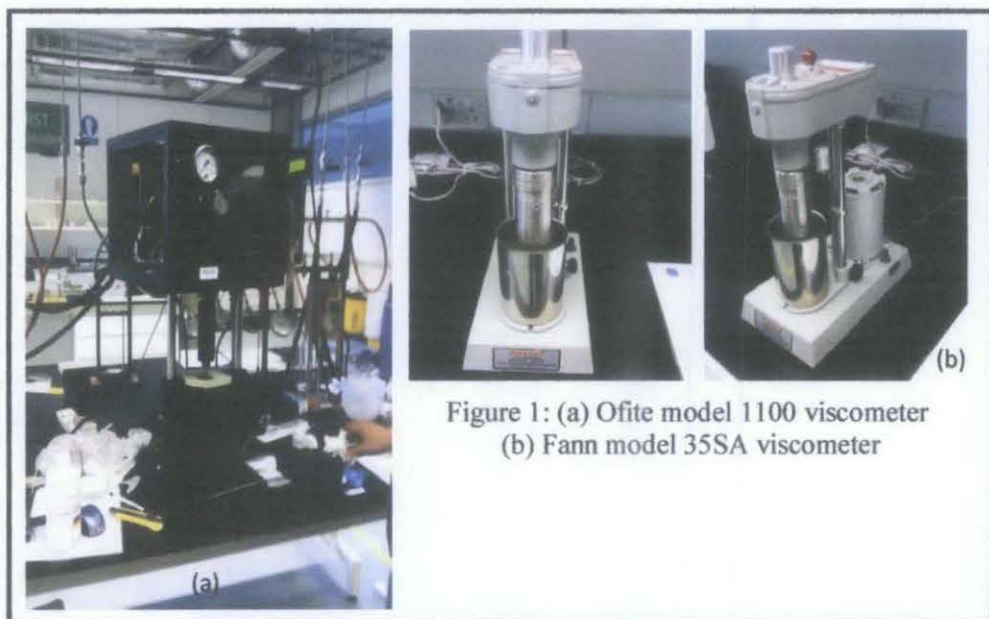
A liquid showing any deviation from the above behaviour is non-Newtonian.

2.3 RHEOMETER AND VISCOMETER

A rheometer is a specialized viscometer that measures visco-elastic properties of materials more than just viscosity. A rheometer, therefore, measures material behaviour such as yield stress, kinetic properties, complex viscosity, modulus, creep, and recovery [1]. The difference between a viscometer and rheometer is basically the components quality and control capabilities [2]. Viscometer is a device in which a spindle rotates in a single direction. Viscometers are limited to be applied to more viscous materials. It is suitable for simple material [2]. Rheometer measure the way in which a liquid, suspension or slurry flows in response to applied forces. It is used for those fluids which cannot be defined by a single value of viscosity and therefore require more parameters to be set and measured than is the case for a viscometer [1].

Most rheometer models belong to three specific categories. These are the rotational rheometer, the capillary rheometer, and the extensional rheometer. The most commonly used of these is the rotational rheometer, which is also called a stress/strain rheometer, followed by the capillary rheometer [3].

A rheometer is used to measure the rheology of semi-solids, suspensions, emulsions, and polymers in industries such as pharmaceuticals, foods, cosmetics, consumer products, and also petrochemical waste. These measurements can help predict shelf life of products under various stress conditions.



Apart from rheological measurements, the wastes are also characterized by using the following equipments:

2.4 CHNS ANALYZER

It was also called as ultimate analysis. CHNS analyzer is an instrument used to determine the elemental composition of sample. The name CHNS is derived from four (4) primary elements measured by the device which are carbon (C), hydrogen (H), nitrogen (N) and sulphur (S) [4]. The samples used in the experiment are solid and semisolid form. Liquid form could not be tested. Before we run the instrument with our samples, sulfamethazine sample should be run first because it acts as a standard reference to other samples.

The samples crushed and weighed (1.5-2 mg) in a tin capsule and then being combusted in a CHNS reactor at approximately 1000°C. The sample, together with the tin capsule and sorbit melts is combusted in enriched oxygen atmosphere. The combustion produces CO₂, SO₂, and NO₂ which then pass through a glass column packed with oxidation catalyst of tungsten trioxide (WO₃) and a copper reducer, kept at 1000°C [4]. The combustion product is reduced to its elemental composition and quantified.

The author's hoping to find important information/data in the experiment as an additional experiment to further characterize the wastes.

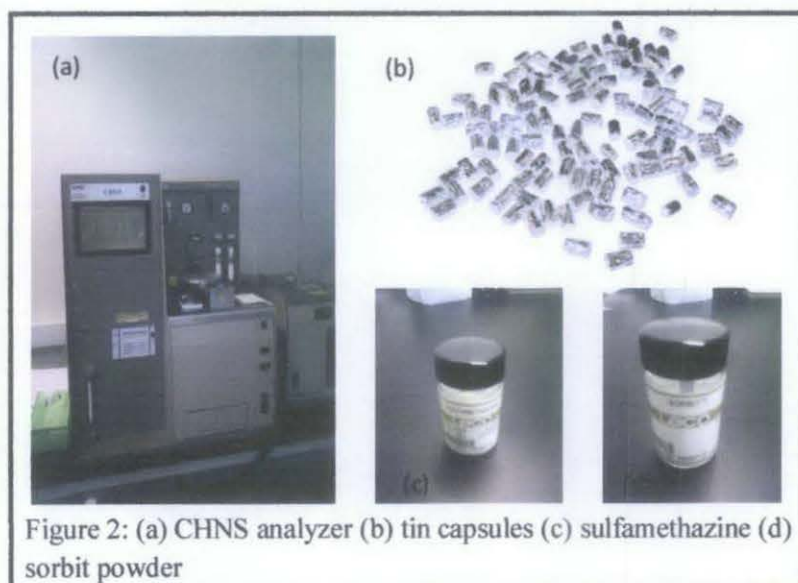


Figure 2: (a) CHNS analyzer (b) tin capsules (c) sulfamethazine (d) sorbit powder

2.5 X-RAY DIFFRACTION



Figure 3: X-Ray Diffraction

According to Scintag, Inc manual chapter 7, X-ray diffraction (XRD) experiment used to reveal the crystallographic structure (the arrangement of atoms within a crystal), chemical composition and physical properties of materials. A powdered sample of material places in a holder, hit by the X-ray beam. The x-ray beam strikes the crystal structure of the sample and causes the beam to spread at specific direction. Only powdered form sample applicable for this experiment.

2.6 THERMO GRAVIMETRIC ANALYSIS



It was also called as proximate analysis. Thermo gravimetric analysis (TGA) measures the amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere [5]. Measurements are used to determine the composition of materials and to predict their thermal stability at temperatures up to 1000°C. TGA can characterize materials that change in term of weight loss or gain due to decomposition, oxidation or dehydration [5].

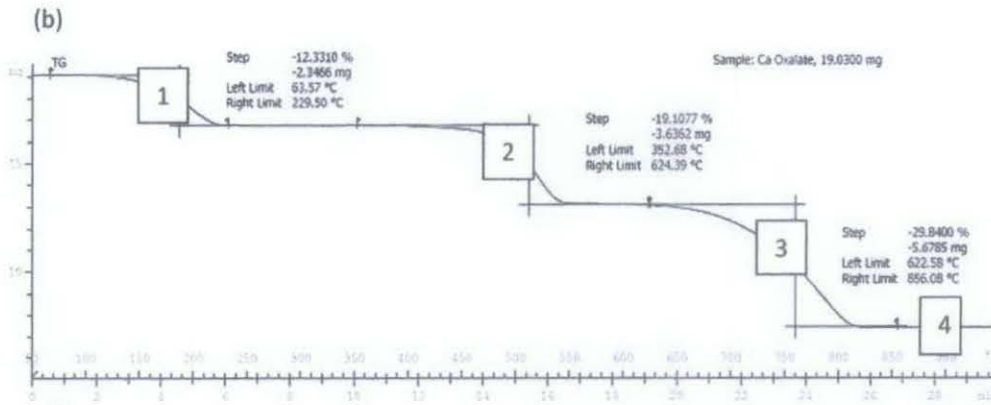


Figure 4: (a) Thermo gravimetric Analysis (b) TGA sample result

The data for TGA are usually presented on dry basis based on its result slopes.

- 1) Moisture content (%) = $100 - m_d$
- 2) Volatile matter (% ,moisture free (mf)*)= $(m_d - m_c) / m_d \times 100$
- 3) Ash content (% ,mf)= $m_f / m_d \times 100$
- 4) Fixed carbon (% ,mf)= $100 - \text{volatile matter (\% ,mf)} - \text{ash content (\% ,mf)}$

*Often expresses as dry

2.7 BOMB CALORIMETER

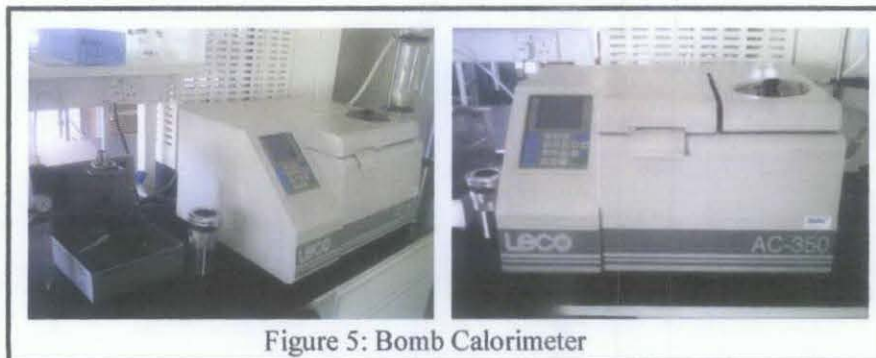


Figure 5: Bomb Calorimeter

A bomb calorimeter is a type of constant-volume calorimeter used in measuring the heat of combustion of a particular reaction. A bomb calorimeter is a sealed container capable of holding several atmospheres of gas pressure. A weighed sample of substance is placed in near contact with an ignition wire inside the bomb. The bomb is pressurised with oxygen, sealed, and placed in the water. An electric current is passed through a wire to ignite the mixture. The data is automatically recorded and save.

2.8 PETROCHEMICAL WASTE

In this project the author's is characterizing the waste from the petrochemical industry. The petrochemical industry is a large and complex industry that is very difficult to define because their operations are intertwined functionally or physically with the inorganic sector of the chemical industry, with downstream, fabrication or compounding activities, or with the petroleum refining industry. The process converts a raw material into product, by-product, intermediate products or waste. There are several types of waste with different forms produced from the petrochemical industry. Some of them were hazardous and dangerous to the environment. Further research and readings is done in choosing the proper petrochemical waste so that it will not harmful to the human being.

Many petrochemical processes are located at or near petroleum refining operations, therefore, many of the air pollutants and hazardous wastes generated by the petroleum industry are also present at petrochemical facilities. According to Federal Energy Administration, Report to congress on Petrochemical, Public Law 93-275, Section 23, waste source of emission can be categorized in 5 ways:

1. Waste containing a principal raw material or product.
2. By-products produced during reaction.
3. Spill, leaks, wash down, vessels cleanouts, or point overflow.
4. Cooling tower and boiler blow down, steam condensate, water treatment wastes and general washing water.
5. Surface runoff.

Disposal of solid waste is a significant problem for the petrochemical industry. Waste solid include water treatment sludge, ashes, fly ash, and incinerator residue, plastics, ferrous and non ferrous metals, catalyst, organic chemicals, inorganic chemicals, filter cake sand viscous solid.

The author's samples of waste are being collected from PETRONAS petrochemical industry, Malaysia Liquefied Natural Gas (MLNG) Bintulu, Sarawak. On 13th September 2010, the author's receive four (4) waste samples which are:

1. Used lubricant oil
2. Charcoal dust
3. Used molecular sieve
4. Sludge Debris

In MLNG, most of the wastes produces were in solid and liquid phases. The wastes are being kept in designated storage at proper place before a designated contractor transports them away. MLNG spend nearly RM 1,000,000 (million) monthly paid to the contractor to manage its waste production. The waste will then be incinerated at the contractor facilities.



Figure 6: used lubrication oil

SAMPLE 1

- Flammability = low
- Toxicity = minimum
- Body contact = moderate
- Reactivity = low
- Chronic = minimum
- Form = liquid
- Color = chocolate



Figure 7: charcoal dust

SAMPLE 2

- Flammability = moderate
- Toxicity = moderate
- Body contact = moderate
- Reactivity = moderate
- Chronic = moderate
- Form = solid (dust)
- Color = black



Figure 8: used molecular sieve

SAMPLE 3

- Flammability = low
- Toxicity = minimum/ nil
- Body contact = minimum/ nil
- Reactivity = low
- Chronic = moderate
- Form = solid
- Color = grey



Figure 9: sludge debris

SAMPLE 4

- Flammability = unknown
- Toxicity = unknown
- Body contact = unknown
- Reactivity = unknown
- Chronic = unknown
- Form = sludge/ semi solid
- Color = black chocolate

2.9 MALAYSIA LIQUEFIED NATURAL GAS SDN BHD

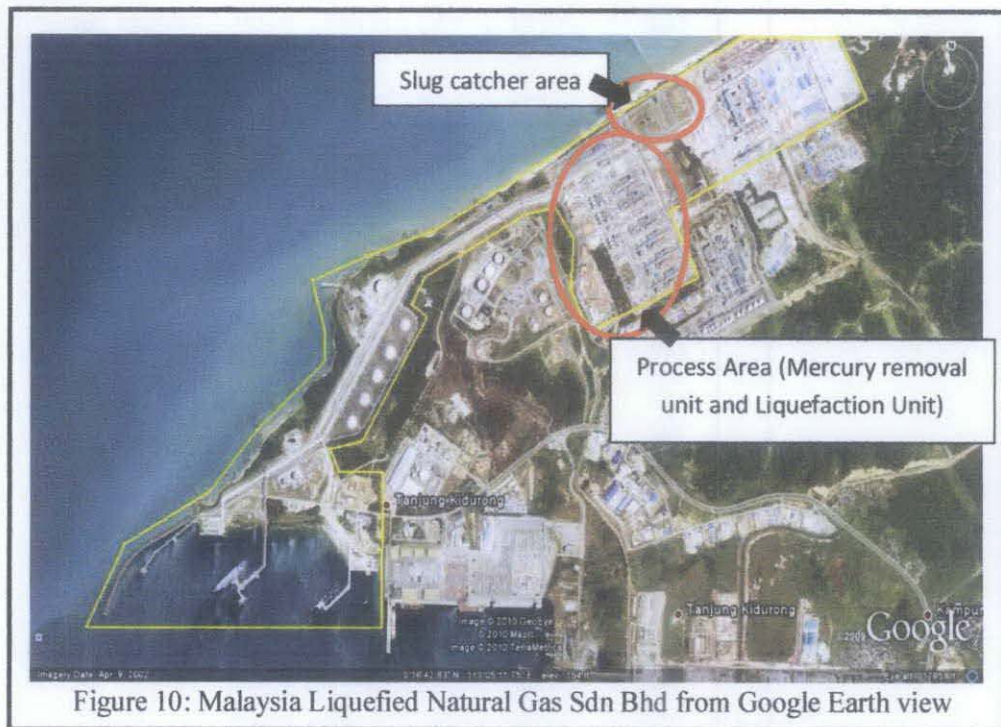


Figure 10: Malaysia Liquefied Natural Gas Sdn Bhd from Google Earth view

Waste	Form/condition	Location in MLNG
Used lubrication oil	Liquid	In rotating equipment, all unit
Used charcoal dust	Solid/powder	Mercury removal unit
Sludge debris	Semisolid	Slug catcher
Molecular sieve	Solid/pallet	Liquefaction unit

Table 1: Form/condition and the location of the waste samples in MLNG area

2.10 LNG PROCESS FLOW

300 to 400 millions of years ago, tiny sea plants and animals died and were buried on the ocean floor and over time they were covered by layers of sand and silt. Over millions of years later, these remains were buried even deeper and the enormous heat and pressure turned them into what we have today as oil and gas [20]. Today, we drill down through these layers of sand, silt and rock until we reach the rock formations that contain oil and gas deposits [20].

Natural gas is an environmental-friendly energy source, consisting of a natural mixture of hydrocarbons, predominantly methane and inert gases which burns cleaner than other fossil fuels. In addition, natural gas emits less carbon dioxide than coal or oil and entails a smaller emission of nitrogen oxide and absolutely no emission of sulphur oxide [20].

In a natural gas liquefaction plant such as MLNG, natural gas which is in gas phase and considered as raw and dirty is converted into liquefaction natural gas (LNG) which is in liquid phase and considered as clean within specifications. The LNG is at atmospheric pressure with temperature of -160°C . In addition the LNG occupies 1600 the volume occupied by natural gas as standard conditions which maximizes the profit gained per unit of energy delivered.

To produce the end product, the natural gas will have to undergo processes in which the main processes are Acid Gas Removal, Dehydration, Mercury Removal, Liquefaction and Fractionation. The diagram below explains briefly the flow of process in an LNG processing module.

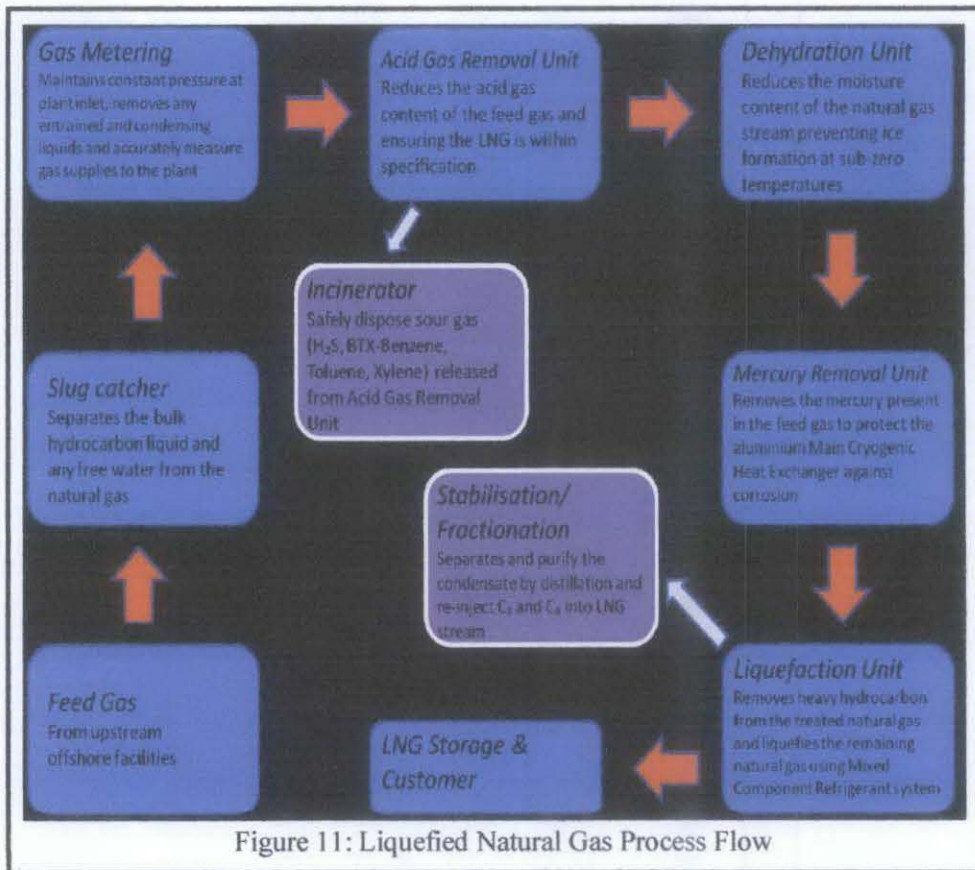
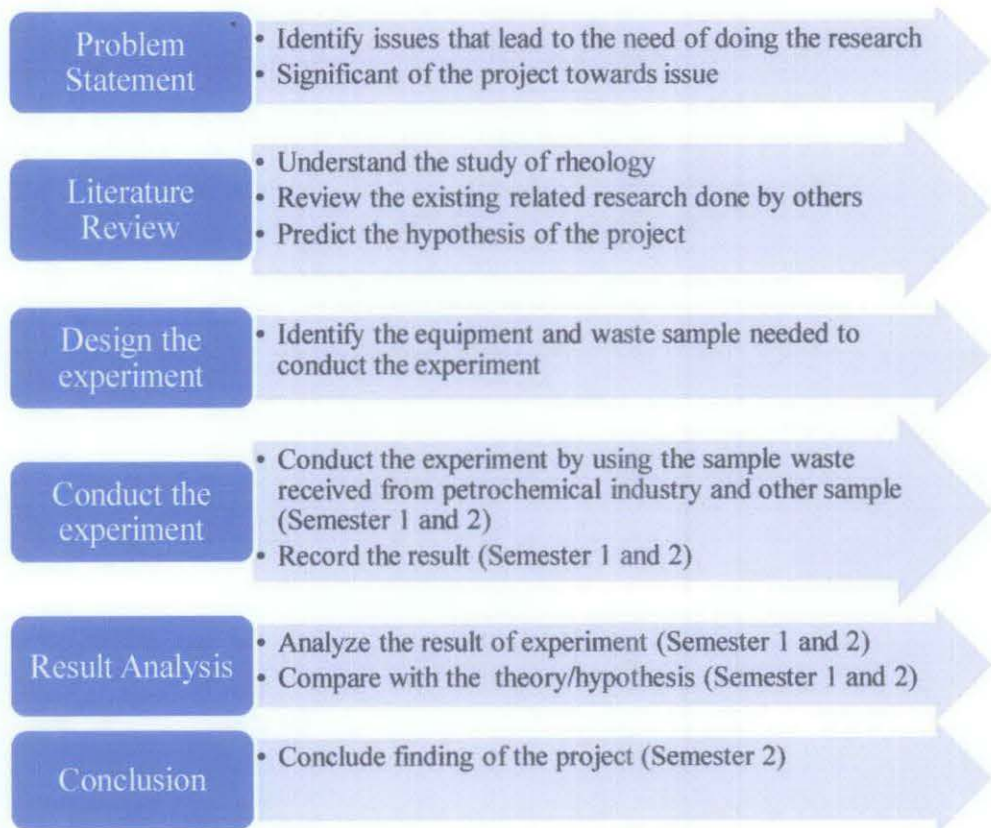


Figure 11: Liquefied Natural Gas Process Flow

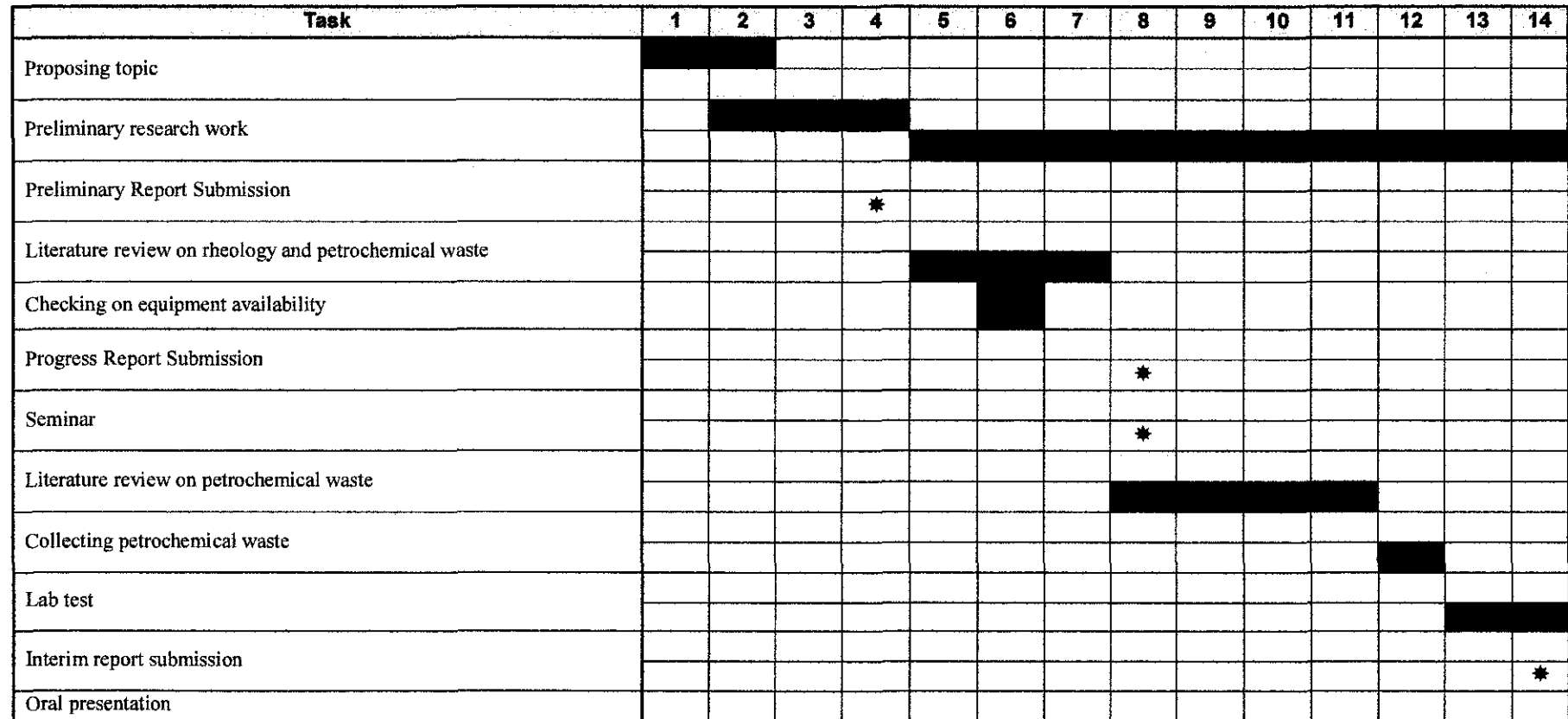
CHAPTER 3

METHODOLOGY

3.1 RESEARCH METHODOLOGY



3.2 PROJECT PLANNING



Legend	
Done	■
Progress	■
Dateline	*

Figure 12: Gantt chart FYP I

Task	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	
Project work continues	Done															
Submission of progress report								*								
Project work continues								Done								
Pre- EDX											*					
Submission of draft report												*				
Submission of dissertation (soft bounded)													*			
Submission of technical paper													*			
Oral presentation														*		
Submission of project Dissertation (Hard bound)															*	

Figure 13: Gantt chart FYP II

Legend	
Done	■
Progress	■
Dateline	*

3.3 EXPERIMENT PLANNING

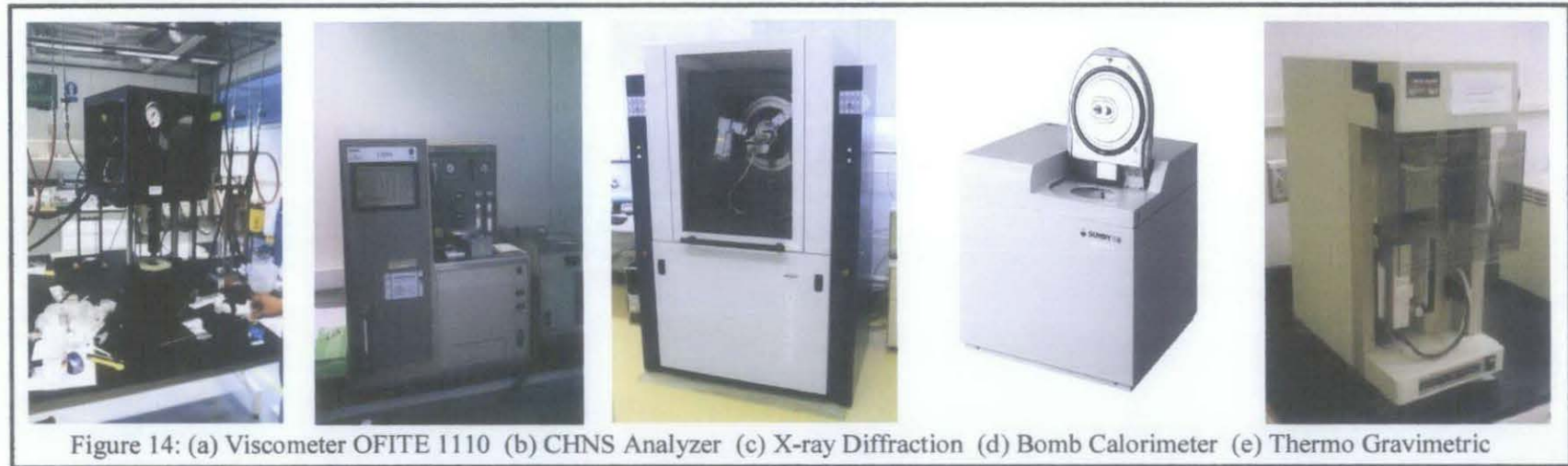


Figure 14: (a) Viscometer OFITE 1110 (b) CHNS Analyzer (c) X-ray Diffraction (d) Bomb Calorimeter (e) Thermo Gravimetric

Waste Samples \ Experiments	Rheometer	CHNS Analyzer	X-Ray Diffraction	Bomb Calorimeter	Thermo gravimetric
Used Lubricant oil (LO)	X			X	
Molecular Sieve (MS)		X		X	X
Charcoal Dust (CD)		X	X	X	X
Sludge Debris (SD)		X		X	X

Table 2: Experiments Compatibility with the Waste Samples

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 VISCOMETER OFITE1110 ANALYSIS (USED LUBRICANT OIL)

The operating condition of the analysis:

<u>1ST RUN ANALYSIS</u>	<u>2ND RUN ANALYSIS</u>
Date: Wed, Jun 08	Date: Wed, Nov 16
Time: 11:28:51	Time: 12:50:15
Length: 165	Length: 22
Shear Stress Unit: Dyne/cm ²	Shear Stress Unit: Pa.
Rate Unit: RPM	Rate Unit: RPM
Temperature Unit: ° C	Temperature Unit: ° C
Pressure Unit: psi	Pressure Unit: psi

Table 3: Viscometer OFITE 1110 Operating Condition

The quantity of Used Lubricant Oil used for each run is 42ml. The first run of the analysis; the author's vary the temperature from 60° C to 30° C at constant RPM 300 and 600. The second run the author's, vary it RPM from 600, 500, 400, 300, 200 and 100 at two (2) different temperatures 40° C and 30° C. This experiment is intended to review the behaviour of the used lubricant oil due to temperature and spindle speed changes.

4.1.1 FIRST TEST RUN RESULTS

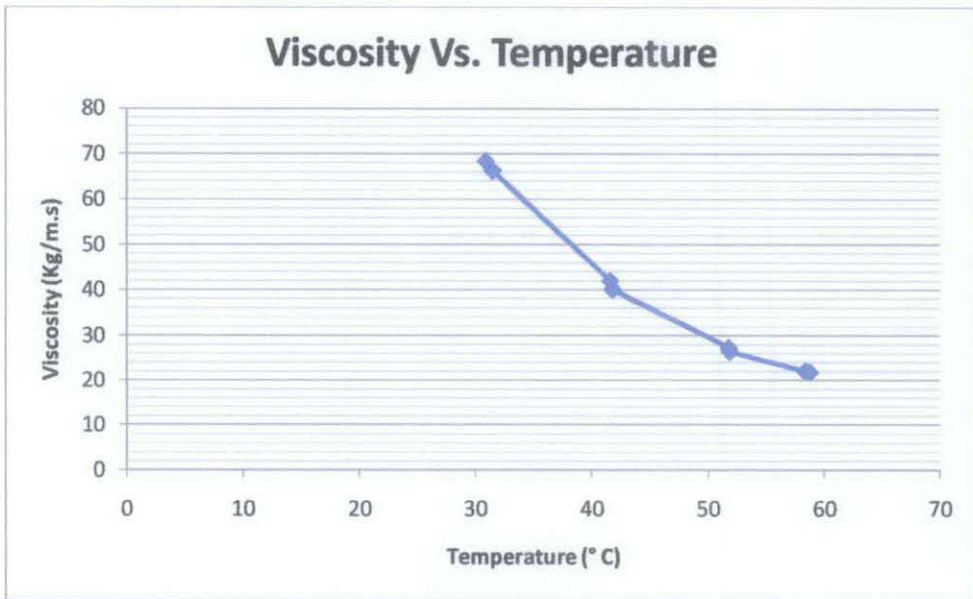


Figure 15: Viscosity by Temperature Variation

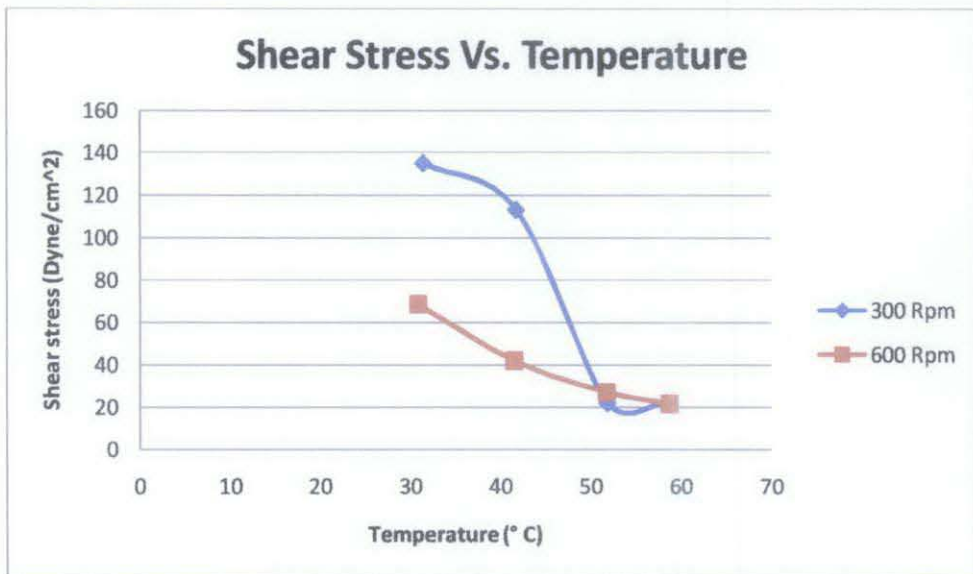


Figure 16: Shear Stress by Temperature variation

4.1.2 SECOND TEST RUN RESULTS

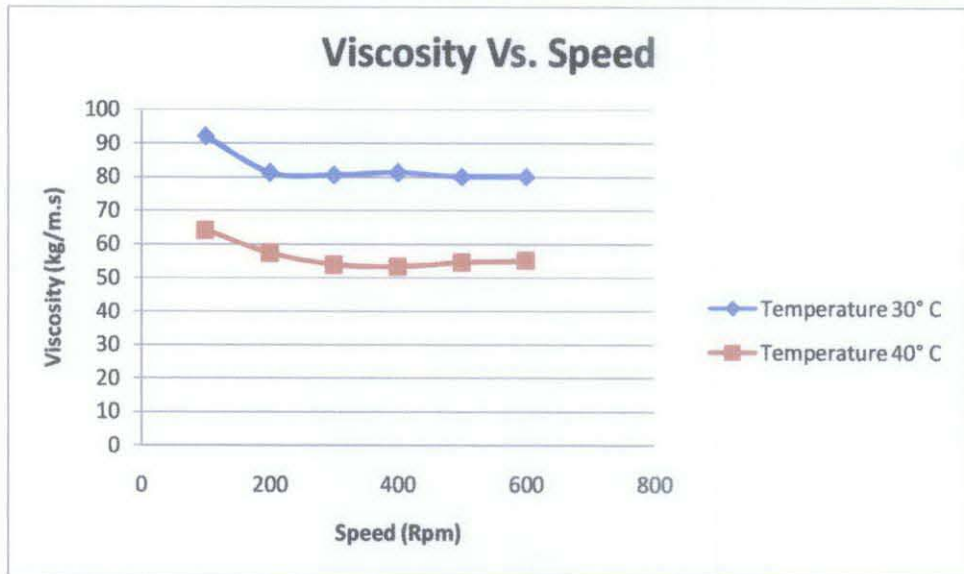


Figure 17: Viscosity by Speed (RPM) variation

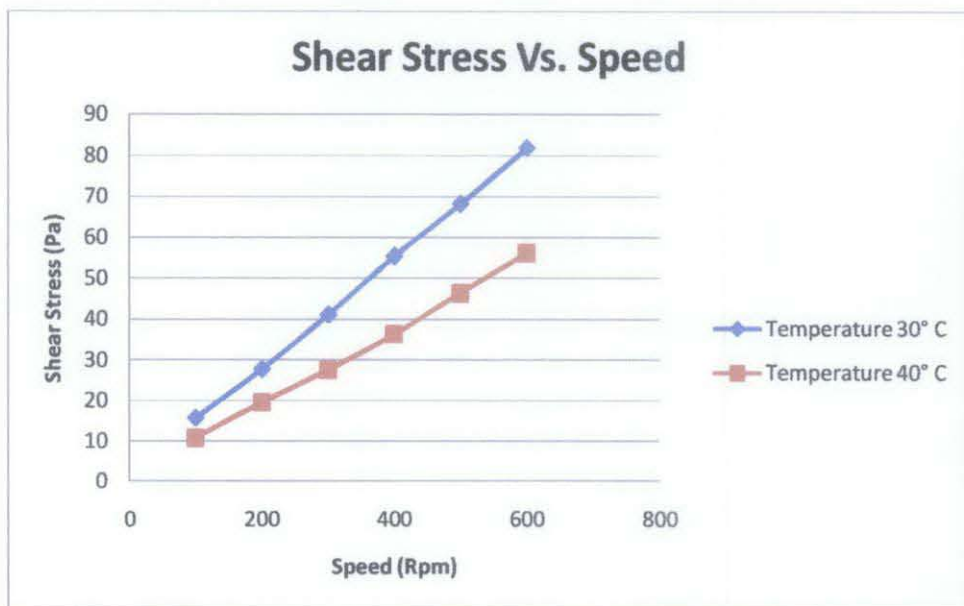


Figure 18: Shear Stress by Speed (RPM) variation

Figure 15 and figure 16 illustrate the effect of varying the temperature at the bath from 90° C to 40° C at constant RPM 300 and 600. The viscosity and shear stress are gradually decreasing as the temperature of the bath increasing.

Figure 17 and figure 18, illustrate the effect of varying the speed at RPM 600, 500, 400,300, 200 and 100 at two (2) different temperatures condition 40° C and 30° C. The viscosity is nearly constant and shear stress are gradually increasing as the RPM increasing while the temperature is maintained at 40° C and 30° C.

From the above figures, the used lubricant oil is in Newtonian category of fluid. The viscosity does not vary with shear stress. However the viscosity of the used lubricant oil affected much by the change in temperature. All data from the figures are included in the Appendix.

This experiment is to check the “pump ability” of the fluid in the process for the possibility of re-refining. It is important to determine the most efficient temperature and pressure for the used lubricant oil to flow in pipe line thus reducing its life cycle cost. Life cycle cost equation:

$$LCC = C_{ic} + C_{in} + C_e + C_o + C_m + C_s + C_{env} + C_d$$

LCC = life cycle cost

C_{ic} = initial costs, purchase price (pump, system, pipe, auxiliary services)

C_{in} = installation and commissioning cost (including training)

C_e = energy costs (predicted cost for system operation, including pump driver, controls, and any auxiliary services)

C_o = operation costs (labor cost of normal system supervision)

C_m = maintenance and repair costs (routine and predicted repairs)

C_s = down time costs (loss of production)

C_{env} = environmental costs (contamination from pumped liquid and auxiliary equipment)

C_d = decommissioning/disposal costs (including restoration of the local environment and disposal of auxiliary services).

For the initial investment cost, C_{ic} , the pump plant designer or manager must decide the outline design of the pumping system. The smaller the pipe and fitting diameters, the lower the cost of installation. However the small diameter installation requires a more powerful pump resulting in higher initial and operating cost [21].

For high viscosity fluid, powerful pump is required to flow. By heating the fluid to reduce its viscosity, we can decide the most reliable and purchase the most efficient pump thus reducing the life cycle cost.

4.2 CHNS ANALYZER OR ULTIMATE ANALYSIS (MOLECULAR SIEVE, CHARCOAL DUST AND SLUDGE DEBRIS)

CHNS analyzer on molecular sieve, charcoal dust and sludge debris are part of standard method to determine the quality of the wastes especially. Sulfamethazine used as a standard chemical for CHNS. All the samples have been weighed with tin capsules. For sludge debris, an additional of sorbit to absorb liquid is required. All samples have been analyzed three (3) times. Our objective is to get the higher percentage of carbon and lowest possible percentage of sulphur and hydrogen. The average CHNS content and its absolute standard deviation for each sample are given below:

	CARBON (%)	HYDROGEN (%)	NITROGEN (%)	SULPHUR (%)
SD01	13.03	2.893	-1.417	3.44
SD02	15.77	3.327	2.682	4.212
SD03	15.93	3.21	2.69	6.995

	CARBON (%)	HYDROGEN (%)	NITROGEN (%)	SULPHUR (%)
MS01	1.788	1.645	0.417	1.498
MS02	1.63	1.751	0.379	0.575
MS03	1.804	1.577	0.43	0.224

	CARBON (%)	HYDROGEN (%)	NITROGEN (%)	SULPHUR (%)
CD01	26.56	1.392	1.181	1.641
CD02	23.98	1.767	1.33	3.42
CD03	36.45	1.556	1.181	1.756

Table 4: The results of CHNS content for Sludge Debris (SD), Molecular Sieve (MS), and Charcoal Dust (CD)

The average CHNS content for each sample:

	CARBON (%)	HYDROGEN (%)	NITROGEN (%)	SULPHUR (%)
SD	14.91	3.14	1.318	4.88
MS	1.74	1.658	0.408	0.7567
CD	29.00	1.572	1.231	2.27

Table 5: The Average results of CHNS content for Sludge Debris (SD), Molecular Sieve (MS), and Charcoal Dust (CD)

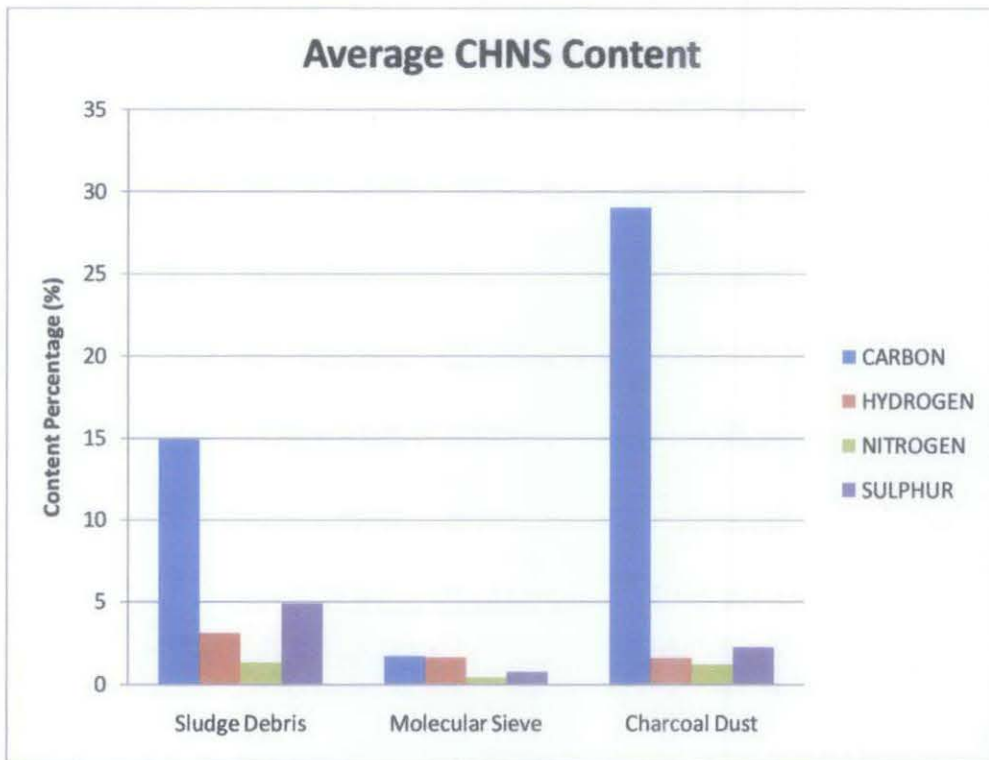


Figure 19: Average CHNS Content for Each Sample

Figure 19 illustrate the average results of CHNS content percentage for each sample. Charcoal dust presents the highest percentages of carbon content of 29% than molecular sieve (1.74%) and sludge debris (14.91%). Charcoal dust probably would have the potential to become energy source based on its carbon content.

Sludge debris presents the highest percentages of hydrogen and sulphur. This results claim that sludge debris toxicity and corrosive ability is higher. Special re-process technique required to overcome the toxicity and the corrosive ability.

Hydrogen and sulphur content for molecular sieve and charcoal dust still within the acceptable ranges.

4.3 X-RAY DIFFRACTION (CHARCOAL DUST)

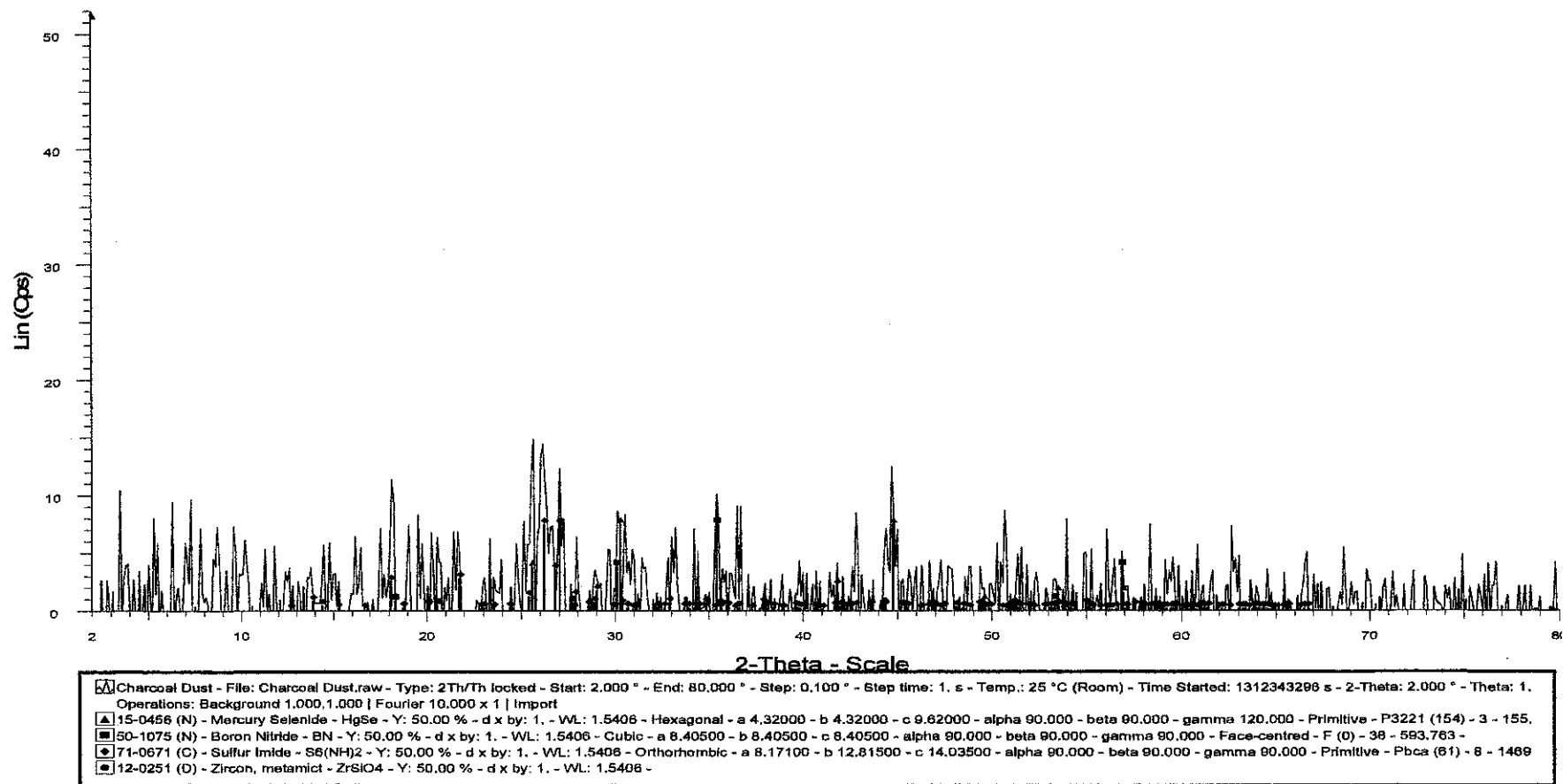


Figure 20: XRD Curve of the Charcoal Dust

The pink colour represent the presence of mercury selenide (HgSe), red for boron nitride (BN), blue for sulphur imide (S6 (NH) 2) and green for zircon, metamict (ZrSiO₄) in the charcoal dust waste sample.

Based on XRD curve of the charcoal dust, figure 23, several diffraction peaks were assigned with mercury selenide (HgSe), boron nitride (BN), sulphur imide (S6 (NH) 2) and zircon, metamict (ZrSiO₄) chemical compositions. According to references, all of the chemical composition were stable and exhibit low toxicity.

According to MLNG chemical watch hazard rating, the charcoal dust shows moderate value of toxicity. The charcoal dust exhibit mercury selenide content because it was used to absorb the mercury from the raw LNG. This result is important to know other composition of properties rather than by its name, to check whether the sample could be harmful to be re-processed based on its component.

4.4 BOMB CALORIMETER (MOLECULAR SIEVE, CHARCOAL DUST AND SLUDGE DEBRIS)

Sample	Heating Value (Kj/g)			Average (Kj/g)
	1	2	3	$\frac{n1+n2+n3}{3}$
Charcoal Dust	17.26	18.69	17.50	17.82
Used Lubricant oil	48.86	45.75	46.29	46.97
Sludge Debris	7.73	7.55	8.04	7.77

Table 6: heating value for Charcoal dust, used lubricant oil and sludge debris

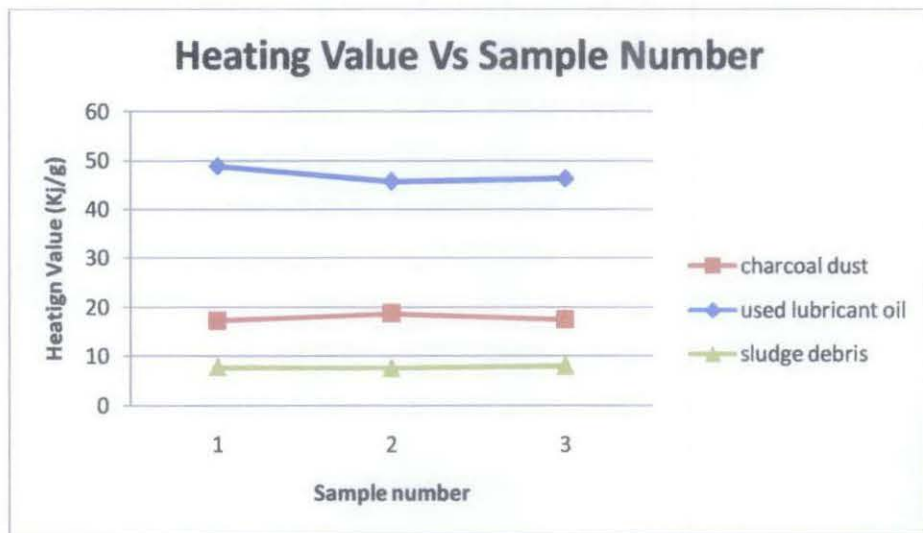


Figure 21: Heating value for each sample versus samples

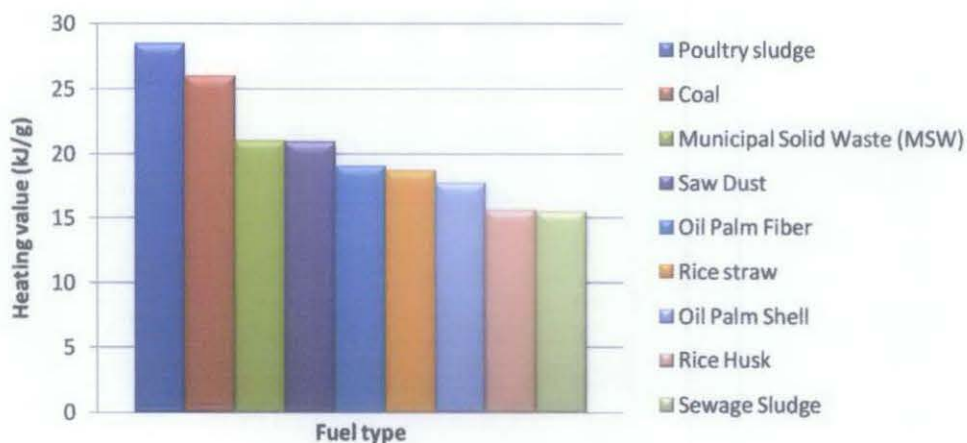


Figure 22: Biomass and coal heating value

By referring from the above results, it shows that the used lubricant oil sample give the highest average heating value (46.97kJ/g) by its combustions compared to the charcoal dust (17.82kJ/g) and sludge debris (7.77 kJ/g). By comparing the heating value of these samples than other kind of biomass and coal type, the heating value of used lubricant oil is too much higher than others (below 30kJ/g). The heating value is higher and can be compared to other types of fuel like diesel, 44.8 kJ/g and petroleum, 43 kJ/g.

The used charcoal dust heating value is slightly lower than usual coal types which is between 30-25 kJ/g and has the same range as oil palm fibre, rice straw and oil palm shell which is in between 20-15 kJ/g. Others heating value is included in the appendix B.

Sludge debris heating value is quite low (7.77kJ/g) and does not appropriate to be re-processed as a fuel. However others characteristic of it should be considered. For the molecular sieve, there is no temperature increment occur. This results show the probability of the samples to be re-processed as a fuel.

4.5 THERMO GRAVIMETRIC OR PROXIMATE ANALYSIS (MOLECULAR SIEVE, CHARCOAL DUST AND SLUDGE DEBRIS)

For thermo gravimetric analysis, the waste samples (approximately 10mg) were carried out at 10 °C/min under N₂ over the whole range of temperature (50 °C – 800 °C) using TGA Q50.

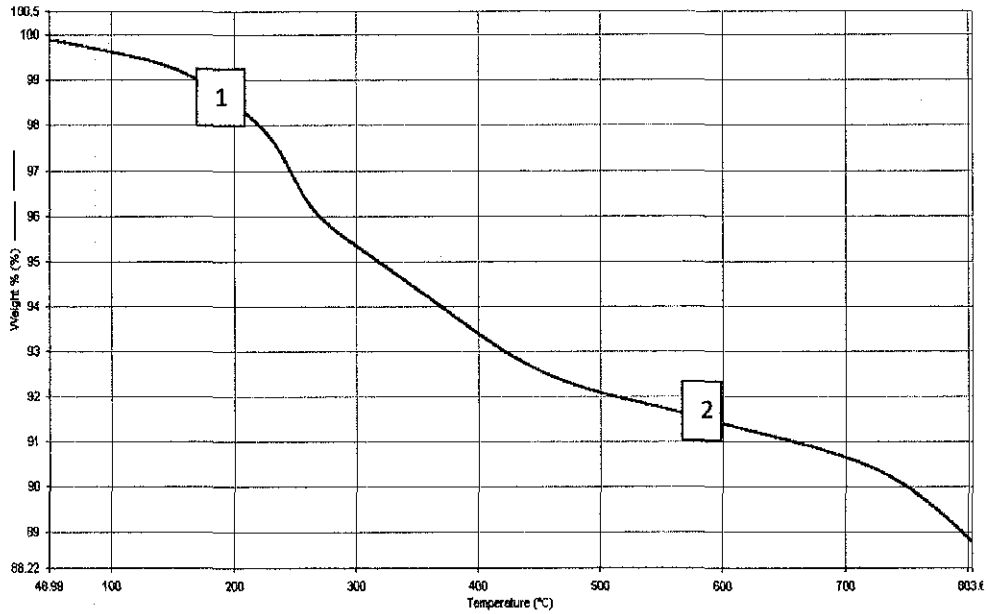


Figure 23: TGA Curve Formulation for Charcoal Dust

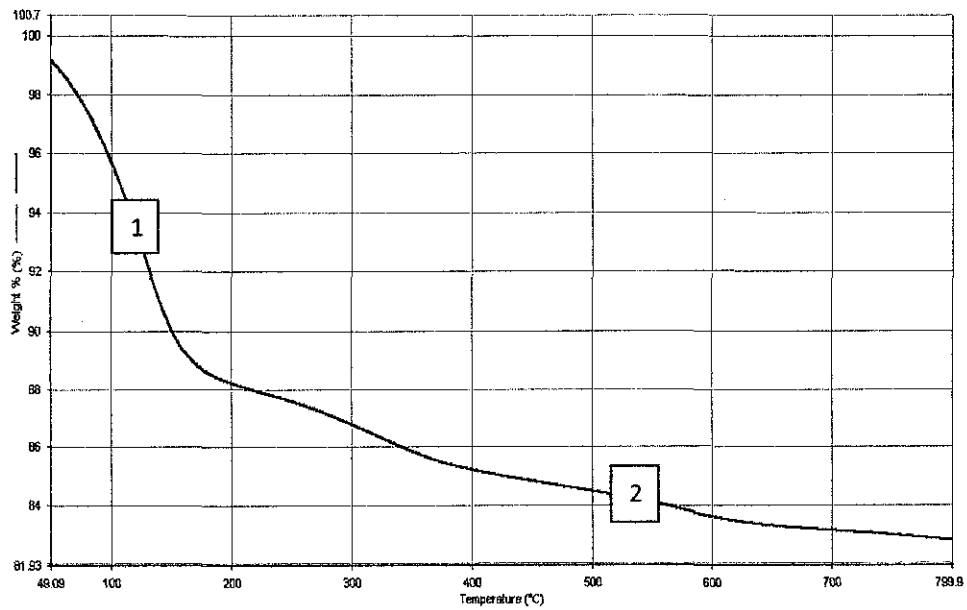


Figure 24: TGA Curve Formulation for Molecular Sieve

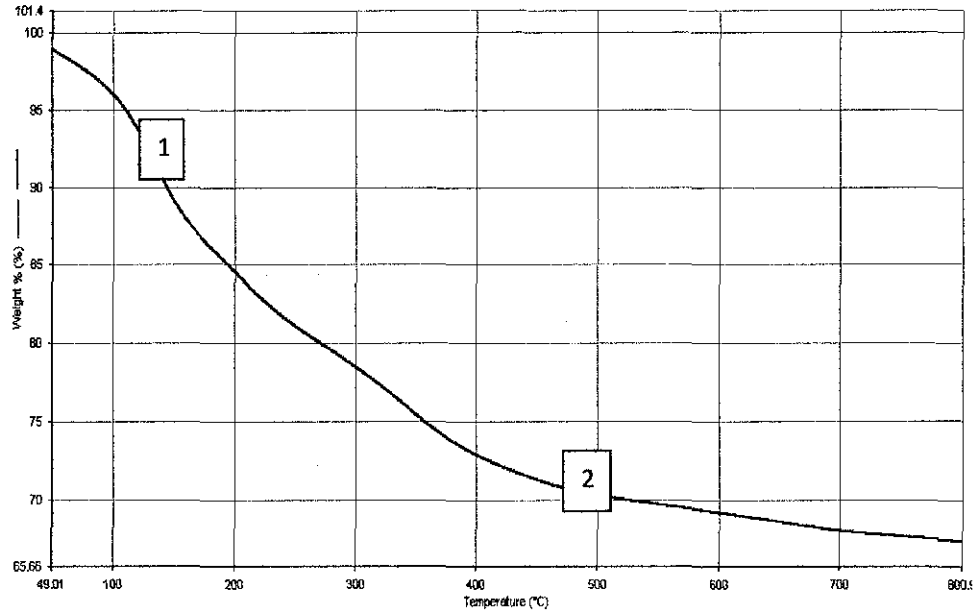


Figure 25: TGA Curve Formulation for Sludge Debris

Based on the TGA results, it was shown the samples weight decreases as the temperature increases. However the samples didn't burn completely as there is still residual weight left although the samples were burn at 800°C. Each sample only exhibit 2 different slopes.

The molecular sieve, as shown in figure 24, shows a drastically decrease in weight percentage at temperature 100°C to 200°C and maintain decreasing at constant rate until 82.78% at temperature 800°C.

The sludge debris, as shown in figure 25, shows an enormous drop of weight percentage until 67.28% at temperature 800°C.

Based from figure 23, figure 24 and figure 25, it was observed that charcoal dust has the higher residual weight percentage (88.78%) at temperature 800°C than molecular sieve and sludge debris. This shows that charcoal dust have thermal stability which may be good for further combustion.

However, the samples should be burn until the residual weight percentages reach zero value to calculate the moisture content, volatile matter, ash content and fixed carbon content. All data were presented in percentage basis. The result is

important to determine its ignition and combustion. Lesser moisture content results in better combustion ability.

CHAPTER 5

CONCLUSION AND RECCOMENDATION

5.2 CONCLUSION

Based on the result and analysis shown in chapter 4, used lubricant oil is a Newtonian type of fluid that the viscosity does not vary with shear stress. Its viscosity is much affected by the temperature changes. Based on its average heating value, the used lubrication oil has the highest 46.97 kj/g and having the same ranges as diesel and petroleum. This high heating value gives a good combustion property. The production rate of the waste is the highest among other waste samples.

Charcoal dust wastes also have the potential to become fuel as it exhibit good heating value 17.82 kj.g and high carbon content at high temperature 14.91%. The charcoal dust contains mercury selenide which has moderate value of toxicity. Further research on charcoal dust toxicity is required to check its capability as a fuel.

Special treatment should be taken for sludge debris because of its high sulphur content. Molecular sieve doesn't exhibit high percentage of sulphur content and doesn't have any temperature increment in bomb calorimeter experiment. The properties of the waste proof that it can be kept safe without harmful to the environment. As a conclusion, used lubricant oil, charcoal dust and sludge debris has a big potential to be reprocess and reused. While the molecular sieve wastes could be safely stored without it being harmful.

This research is successfully done to identify the possible waste that could be re-processed as useful materials for example a fuel. By having this research, we can reduce the total of waste being dumped away without any benefits. The benefits from these re-processed wastes can be used in hoping to reduce the cost of managing the waste and increasing the company profit in the future.

5.2 RECCOMENDATION

1. Continue research on used lubricant oil that has the potential to be reused by using the re-refining process techniques back to its original condition. Also it has the capability as a fuel based on its heating value. Easy to re-process due to it Newtonian fluid type characteristic.
2. More research and experiment needs to be done on the charcoal dust and sludge debris from LNG plant because of its high potential to be reused. The toxicity contain in each samples should be considered during processing.
3. Enhance the research by using more experiment type to improve the data available.
4. Run the thermo gravimetric analysis until the residual weight percentages for each sample reach zero.
5. More comparison with other existence re-processing techniques to check the compatibility.
6. Early booking of lab is required to avoid unavailability and error.
7. More collaboration with petrochemical industry during research to understand the history of the waste samples because it's important during characterization of the waste.

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APPENDIX A

First test run log data, the temperature varies from 90° C to 40° C at constant speed (RPM) 300 and 600.

Log Data:

Elapsed Time	Temperature	Shear Stress	Viscosity	RPM	Shear Rate	Pressure	Bath Temp
00:00.0	26.3	0	0	0	0	-0.3	24.6
00:26.1	26.3	436.6	85.5	300	510.69	0.4	28.4
01:26.1	26.4	419.9	82.2	300	510.69	0.1	50.1
02:26.1	27	374.1	73.2	300	510.69	0.2	73.2
03:26.2	28.7	316.6	62	300	510.69	0.3	83.4
04:26.2	31.4	272.3	53.3	300	510.69	0.3	82.8
05:26.3	34.5	244.3	47.8	300	510.69	0.5	80.7
06:26.3	37.6	223.7	43.8	300	510.69	-0.1	78.8
07:26.1	40.4	204.5	40	300	510.69	0	77
08:26.1	42.9	190.2	37.3	300	510.69	-0.1	75.4
09:26.1	45	179.2	35.1	300	510.69	0	73.9
10:26.2	46.9	168.8	33	300	510.69	0.1	72.7
11:26.2	48.6	161	31.5	300	510.69	-0.2	71.6
12:26.3	49.9	154.3	30.2	300	510.69	-0.5	70.6
13:26.3	51.2	147.3	28.8	300	510.69	0.1	69.8
14:26.0	52.2	142.6	27.9	300	510.69	0	69.1
15:26.1	53.1	138.3	27.1	300	510.69	-0.5	68.5
16:26.1	53.9	134.4	26.3	300	510.69	0.2	68
17:26.2	54.6	130.7	25.6	300	510.69	-0.2	69.7
18:26.2	55.3	127.4	25	300	510.69	-0.1	72.9
19:26.3	56	122.7	24	300	510.69	-0.2	74.7
20:26.3	56.9	118.6	23.2	300	510.69	-0.4	74.7
21:26.1	57.8	114.3	22.4	300	510.69	-0.2	74.1
22:26.3	58.7	223.7	21.9	600	1021.38	-0.5	73.6
22:39.3	58.9	222.8	21.8	600	1021.38	-0.8	73.5
23:26.1	59.5	109.1	21.4	300	510.69	-0.1	72.6
24:26.1	60.1	107.1	21	300	510.69	-1	71.1
25:26.2	60.6	105.8	20.7	300	510.69	-0.9	69.8
26:26.2	60.9	105.3	20.6	300	510.69	-0.3	68.7
27:26.2	61.2	104	20.4	300	510.69	-0.2	67.6
28:26.3	61.3	104.1	20.4	300	510.69	-0.7	66.6
29:26.3	61.3	103.5	20.3	300	510.69	-0.8	65.7

30:26.1	61.3	103	20.2	300	510.69	0.1	64.8
31:26.1	61.2	104.6	20.5	300	510.69	-0.4	64
32:26.2	61.1	104	20.4	300	510.69	-0.8	63.3
33:26.2	60.9	103.9	20.4	300	510.69	-1.2	62.6
34:26.2	60.7	104.6	20.5	300	510.69	-1.4	61.9
35:26.3	60.5	105.1	20.6	300	510.69	-0.9	61.3
36:26.3	60.3	105.3	20.6	300	510.69	-1.1	60.7
37:26.3	60	106.8	20.9	300	510.69	-1.1	60.1
38:26.1	59.7	106.8	20.9	300	510.69	-1.2	59.6
39:26.1	59.4	107.3	21	300	510.69	-0.8	59.1
40:26.2	59.2	108.8	21.3	300	510.69	-1.1	58.6
41:26.2	58.9	109	21.4	300	510.69	-1.3	58.1
42:26.2	58.5	109.7	21.5	300	510.69	-1.4	57.6
43:26.3	58.2	111	21.7	300	510.69	-1.2	57.1
44:26.4	57.9	113.1	22.1	300	510.69	-1.3	56.7
45:26.1	57.6	112.9	22.1	300	510.69	-1.2	56.3
46:26.1	57.3	114.2	22.4	300	510.69	-1.5	55.9
47:26.2	57	115	22.5	300	510.69	-1.5	55.5
48:26.2	56.7	116.4	22.8	300	510.69	-1.9	55.1
49:26.3	56.4	116.9	22.9	300	510.69	-1.6	54.7
50:26.3	56.1	119.1	23.3	300	510.69	-1.5	54.3
51:26.1	55.8	120	23.5	300	510.69	-2.1	54
52:26.1	55.4	120.3	23.5	300	510.69	-1.7	53.6
53:26.1	55.1	121.6	23.8	300	510.69	-2.1	53.3
54:26.2	54.8	122.7	24	300	510.69	-1.6	52.9
55:26.2	54.6	123.5	24.2	300	510.69	-1.5	52.6
56:26.3	54.3	125.2	24.5	300	510.69	-1.6	52.3
57:26.3	54	126.5	24.8	300	510.69	-1.5	51.9
58:26.3	53.7	127.2	24.9	300	510.69	-1.9	51.6
59:26.1	53.4	128	25.1	300	510.69	-2.4	51.3
00:26.1	53.1	129.2	25.3	300	510.69	-1.1	51
01:26.2	52.9	130	25.5	300	510.69	-2	50.7
02:26.2	52.6	132.1	25.9	300	510.69	-2.6	50.5
03:26.2	52.3	133	26	300	510.69	-1.3	50.1
04:26.3	52.1	133.9	26.2	300	510.69	-2.9	49.9
05:26.2	51.8	272.1	26.6	600	1021.38	-2.4	49.6
05:46.2	51.8	278.8	27.3	600	1021.38	-1.6	49.6
06:26.3	51.6	142	27.8	300	510.69	-1.7	49.4
07:26.1	51.3	142.3	27.9	300	510.69	-1.9	49.1
08:26.1	51.1	143.6	28.1	300	510.69	-2.6	48.9
09:26.1	50.8	144.9	28.4	300	510.69	-2.4	48.6
10:26.2	50.6	146.2	28.6	300	510.69	-2.1	48.4
11:26.2	50.4	146.9	28.8	300	510.69	-2.8	48.1
12:26.3	50.1	148	29	300	510.69	-2.2	47.9
13:26.3	49.9	148.9	29.2	300	510.69	-2.1	47.6

14:26.3	49.7	149.5	29.3	300	510.69	-2.7	47.4
15:26.1	49.5	151.4	29.6	300	510.69	-2.8	47.2
16:26.1	49.3	152.1	29.8	300	510.69	-2.4	47
17:26.2	48.9	156.6	30.7	300	510.69	-1.6	46.6
18:26.2	48.5	160.7	31.5	300	510.69	-2.6	46.3
19:26.2	47.9	164.5	32.2	300	510.69	-2.5	46
20:26.3	47.3	168.2	32.9	300	510.69	-2.4	45.7
21:26.3	46.7	171.9	33.7	300	510.69	-2.7	45.4
22:26.1	46.1	175.7	34.4	300	510.69	-2.2	45.2
23:26.1	45.5	179	35.1	300	510.69	-2.1	44.9
24:26.1	45	182.8	35.8	300	510.69	-2.2	44.6
25:26.2	44.5	186.1	36.5	300	510.69	-2.6	44.4
26:26.2	44	189.7	37.1	300	510.69	-2.6	44.1
27:26.2	43.5	193	37.8	300	510.69	-2.7	43.9
28:26.3	43	197	38.6	300	510.69	-2.7	43.6
29:26.3	42.6	199.8	39.1	300	510.69	-2.3	43.4
30:26.1	42.1	202.7	39.7	300	510.69	-2.2	43.1
31:26.1	41.8	403.3	39.5	600	1021.38	-1.9	42.9
31:46.9	41.6	428.9	42	600	1021.38	-2.5	42.8
32:26.1	41.4	214.8	42.1	300	510.69	-2.5	42.6
33:26.1	41	217.1	42.5	300	510.69	-2.6	42.4
34:26.2	40.7	219.6	43	300	510.69	-2.3	42.2
35:26.2	40.3	222.4	43.5	300	510.69	-2.5	42
36:26.2	40	224.6	44	300	510.69	-1.3	41.7
37:26.3	39.7	227	44.4	300	510.69	-2	41.5
38:26.3	39.4	229.3	44.9	300	510.69	-2.8	41.3
39:26.1	39.1	231.9	45.4	300	510.69	-2.6	41.1
40:26.1	38.8	234.1	45.8	300	510.69	-2.7	40.9
41:26.1	38.6	236.3	46.3	300	510.69	-2.1	40.6
42:26.2	38.4	238.3	46.7	300	510.69	-2.3	40.5
43:26.2	38.1	240.4	47.1	300	510.69	-2.3	40.3
44:26.3	37.9	242.6	47.5	300	510.69	-1.6	40
45:26.3	37.7	244.6	47.9	300	510.69	-2.7	39.8
46:26.1	37.4	246.5	48.3	300	510.69	-2.7	39.6
47:26.1	37.2	248.4	48.6	300	510.69	-2.8	39.4
48:26.1	37	250.5	49.1	300	510.69	-2.4	39.3
49:26.2	36.8	252.3	49.4	300	510.69	-2.6	39.1
50:26.2	36.7	254.1	49.7	300	510.69	-3.1	38.9
51:26.3	36.5	255.2	50	300	510.69	-2.4	38.7
52:26.3	36.4	256.9	50.3	300	510.69	-2.6	38.6
53:26.1	36.2	258.5	50.6	300	510.69	-2.6	38.4
54:26.1	36	259.8	50.9	300	510.69	-2.7	38.2
55:26.1	35.9	261.8	51.3	300	510.69	-3	38
56:26.2	35.8	263.3	51.6	300	510.69	-3.1	37.9
57:26.2	35.6	265	51.9	300	510.69	-2.1	37.7

58:26.3	35.5	266.5	52.2	300	510.69	-3	37.6
59:26.3	35.4	268	52.5	300	510.69	-2.3	37.4
00:26.3	35.3	269.3	52.7	300	510.69	-2.8	37.2
01:26.1	35.2	271.5	53.2	300	510.69	-2.3	37.1
02:26.1	35.1	272.6	53.4	300	510.69	-2.9	36.9
03:26.2	34.9	274	53.7	300	510.69	-2.3	36.7
04:26.2	34.9	274.5	53.7	300	510.69	-3.1	36.6
05:26.3	34.8	276	54	300	510.69	-3	36.5
06:26.3	34.7	276.9	54.2	300	510.69	-2.9	36.3
07:26.3	34.6	277.9	54.4	300	510.69	-2.7	36.2
08:26.1	34.5	278.3	54.5	300	510.69	-3	36
09:26.1	34.4	279.5	54.7	300	510.69	-2.7	35.9
10:26.2	34.3	280.5	54.9	300	510.69	-2.1	35.8
11:26.2	34.3	281.2	55.1	300	510.69	-2.4	35.7
12:26.2	34.2	282.4	55.3	300	510.69	-2.8	35.5
13:26.3	34.1	282.8	55.4	300	510.69	-2.6	35.4
14:26.3	34.1	283.4	55.5	300	510.69	-3	35.3
15:26.3	34	283.9	55.6	300	510.69	-3.9	35.1
16:26.1	33.9	284.9	55.8	300	510.69	-3.1	35
17:26.1	33.9	285.9	56	300	510.69	-2.4	34.9
18:26.2	33.9	286.4	56.1	300	510.69	-2.4	34.8
19:26.2	33.8	287.1	56.2	300	510.69	-2.6	34.7
20:26.2	33.7	287.1	56.2	300	510.69	-2.6	34.5
21:26.3	33.7	288.2	56.4	300	510.69	-2.7	34.4
22:26.3	33.6	288.7	56.5	300	510.69	-3.2	34.3
23:26.1	33.6	288.5	56.5	300	510.69	-2.8	34.2
24:26.1	33.6	289.1	56.6	300	510.69	-3	34.1
25:26.1	33.5	293.8	57.5	300	510.69	-3	33.9
26:26.2	33.3	293.5	57.5	300	510.69	-3.6	33.8
27:26.2	33.2	293.8	57.5	300	510.69	-3.5	33.7
28:26.3	33.1	294.4	57.6	300	510.69	-2.7	33.6
29:26.3	33.1	295.2	57.8	300	510.69	-2.8	33.5
30:26.1	33.1	295.3	57.8	300	510.69	-2.6	33.4
31:26.1	33	295.9	57.9	300	510.69	-3.1	33.3
32:26.1	33	296.3	58	300	510.69	-2.7	33.2
33:26.2	33	296.7	58.1	300	510.69	-3.8	33.1
34:26.2	32.9	296.9	58.1	300	510.69	-3.6	33
35:26.3	32.9	298.1	58.4	300	510.69	-3	32.9
36:26.3	32.9	308.2	60.4	300	510.69	-2.7	32.7
37:26.1	32.5	321.3	62.9	300	510.69	-2.7	32.5
38:26.1	31.7	339.8	66.5	300	510.69	-2.4	32.5
39:12.7	30.9	698.6	68.4	600	1021.38	-3	32.4

Second test run log data; vary its speed (RPM) from 600, 500, 400,300, 200 and 100 at two (2) different temperatures 40° C and 30° C.

Log Data:

Elapsed Time	Temperature	Shear Stress	Viscosity	RPM	Shear Rate	Pressure	Bath Temp
00:00.0	29.4	0	0	0	0	-1.1	33.7
00:10.2	29.4	48.2	0	600	1021.38	-1.3	33.8
01:10.3	29.8	82.3	80.6	600	1021.38	-1.1	38
02:10.3	30.2	55.8	81.9	400	680.92	-1.3	39.5
03:10.3	30.8	27.9	81.9	200	340.46	-1.8	39.6
04:00.4	31.3	15.6	91.9	100	170.23	-1.1	39.5
04:10.3	31.4	52.1	91.9	600	1021.38	-1.5	39.6
05:10.3	32	74.2	72.6	600	1021.38	-1.6	44.8
06:10.1	32.7	71.7	70.2	600	1021.38	-0.6	46.6
07:10.1	33.5	69.5	68.1	600	1021.38	-2	46.4
08:10.2	34.3	68	66.6	600	1021.38	-1.3	45.9
09:10.2	35.1	66.6	65.2	600	1021.38	-0.8	45.3
10:10.2	35.8	64.9	63.6	600	1021.38	-1.2	44.9
11:10.3	36.5	63.4	62	600	1021.38	-1.2	46.3
12:10.3	37.1	61.5	60.2	600	1021.38	-1.4	49.2
13:10.1	37.7	59.4	58.2	600	1021.38	-0.8	51
14:10.1	38.4	57.6	56.4	600	1021.38	-1.6	51.6
15:10.3	39.2	51.5	54.9	500	851.15	-1.5	51.3
16:10.3	39.9	32	52.9	300	510.69	-1.7	50.9
17:10.3	40.6	15.9	57.2	100	170.23	-1.2	50.4
17:36.7	40.9	10.9	64	100	170.23	-0.9	50.2

APPENDIX B

Heating value of other fuels available:

Fuel	Higher Calorific Value (Gross Calorific Value - GCV)	
	<i>KJ/kg</i>	<i>Btu/lb</i>
Acetone	29,000	
Alcohol, 96%	30,000	
Anthracite	32,500 - 34,000	14,000 - 14,500
Bituminous coal	17,000 - 23,250	7,300 - 10,000
Butane	49,510	20,900
Carbon	34,080	
Charcoal	29,600	12,600
Coal	15,000 - 27,000	6,000 - 14,000
Coke	28,000 - 31,000	12,000 - 13,600
Diesel	44,800	19,300
Ethanol	29,700	12,800
Ether	43,000	
Gasoline	47,300	20,400
Glycerin	19,000	
Hydrogen	141,790	61,000
Lignite	16,300	7,000
Methane	56,530	
Oils, vegetable	39,000 - 48,000	
Peat	13,600 - 20,500	5,500 - 8,800
Petrol	48,000	
Petroleum	43,000	
Propane	50,350	
Semi anthracite	26,700 - 32,500	11,500 - 14,000
Sulfur	9,200	
Tar	36,000	
Turpentine	44,000	
Wood, (dry)	14,400 - 17,400	6,200 - 7,500
	<i>KJ/m³</i>	<i>Btu/ft³</i>
Acetylene	56,000	
Butane C ₄ H ₁₀	133,000	3200
Hydrogen	13,000	
Natural gas	43,000	950 - 1150
Methane CH ₄	39,820	
Propane C ₃ H ₈	101,000	2550
Town gas	18,000	
	<i>KJ/l</i>	<i>Btu/imp gal</i>
Gas oil	38,000	164,000
Heavy fuel oil	41,200	177,000
Kerosene	35,000	154,000

- $1 \text{ kJ/kg} = 1 \text{ J/g} = 0.4299 \text{ Btu/lb}_m = 0.23884 \text{ kcal/kg}$
- $1 \text{ Btu/lb}_m = 2.326 \text{ kJ/kg} = 0.55 \text{ kcal/kg}$
- $1 \text{ kcal/kg} = 4.1868 \text{ kJ/kg} = 1.8 \text{ Btu/lb}_m$
- $1 \text{ dm}^3 \text{ (Liter)} = 10^{-3} \text{ m}^3 = 0.03532 \text{ ft}^3 = 1.308 \times 10^{-3} \text{ yd}^3 = 0.220 \text{ Imp gal (UK)}$
 $= 0.2642 \text{ Gallons (US)}$