Anaerobic Treatment of Pharmaceutical Wastewater

by

Muhamad Farhan bin Abd Rahim

Dissertation submitted in partial fulfillment of

the requirements for the

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(Civil Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the

Civil Engineering Programme

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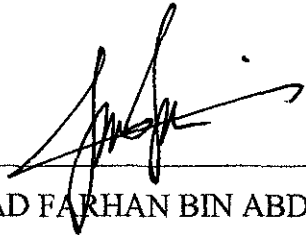
(CIVIL ENGINEERING)

Approved by,

_____
(Assoc Prof Dr Mohamed Hasnain Isa)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
January 2008

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 acknowledgement, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MUHAMAD FARHAN BIN ABD RAHIM

ABSTRACT

This final report is prepared to report the whole progress of the studies of anaerobic treatment of pharmaceutical wastewater. The project analysis done focused on monitoring the performance of upflow anaerobic sludge blanket (UASB) reactor to treat the pharmaceutical wastewater. Two reactors with different operating temperatures, mesophilic ($35\pm1^{\circ}$C) and thermophilic ($54\pm1^{\circ}$C) were used. The sludge chosen for anaerobic reactors was taken from UTP Sewage Treatment Plant. The aim of this project is to determine the optimum operating condition of the reactors and also to monitor the performance of reactors in removing the COD with application of different Organic Loading Rates (OLR) and Hydraulic retention times (HRT). The pharmaceutical wastewater was divided into two strength based on COD concentrations which, low strength (300-500 mg/L) and high strength (1700-2000 mg/L). Based on wastewater strength and the hydraulic retention time, the period of the works is divided by five phase with different OLR ranges. The OLR was increase from 0.07 to 0.56 kg COD/m^3d. The hydraulic retention times (HRT) was ranged from 5 to 3 days. The reactor performance was monitored for 211 days. Phase 1 to 2 consist of low strength wastewater while Phase 3 to 5 consist of high strength of wastewater. The highest percentage of COD removal for mesophilic reactor was 95% while for thermophilic reactor, the percentage removal was 93%. The methane productions occur at range of 50-300 mL for both of the reactors. The Phosphorus for both effluents was 10-40 mg/L. The Ammonia Nitrogen for both effluents was 5-14 mg/L. During phase 4 the reactor failure incident occurs at mesophilic reactor causing its instability to remove COD. Anaerobic reactor has high percentage of COD removal efficiency and the methane produced can be beneficial to industry.

ACKNOWLEDGEMENTS

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CHAPTER 1

INTRODUCTION

1.0   OVERVIEW AND PROBLEM STATEMENT

Production of pharmaceutical products can be broken down into three main stages: (1) research and development; (2) the conversion of organic and natural substances into bulk pharmaceutical substances or ingredients through fermentation, extraction, and/or chemical synthesis; and (3) the formulation and assembly of the final pharmaceutical product.

The discovery of a variety of pharmaceuticals in surface, ground, and drinking waters around the country is raising concerns about the potentially adverse environmental consequences of these contaminants. Minute concentrations of chemicals known as endocrine disruptors, some of which are pharmaceuticals, have detrimental effects on aquatic species and possibly on human health and development. The consistent increase in the use of potent pharmaceuticals, driven by both drug development and our aging population, is creating a corresponding increase in the amount of pharmaceutical waste generated. Pharmaceutical waste is not one single waste stream, but many distinct waste streams that reflect the complexity and diversity of the chemicals that comprise pharmaceuticals. Pharmaceutical waste is potentially generated through a wide variety of activities in a health care facility, including but not limited to intravenous (IV) preparation, general compounding, spills/breakage, partially used vials, syringes, unused preparations, unused unit dose repacks, patients' personal medications and outdated pharmaceuticals. (Oktem, et al., 2007)

The wastewater may therefore be high in biochemical oxygen demand (BOD), chemical oxygen demand (COD), and total suspended solids (TSS), with wide range of pH from 1 to 11 (EPA, 1997). The wastewater is not suitable for physical and/or chemical treatment because of their low efficiency for dissolved COD removal, high consumption of chemicals an high sludge production (Mayabhate et al., 1988). The high concentration of COD in pharmaceutical wastewater makes them potential candidates for anaerobic technology.

Oestrogens and other endocrine disrupting substances are common components of pharmaceutical wastewater. Their effects on wildlife are well documented and there is increasing concern on human health. They have been implicated in testicular cancer, breast cancer, sex organ malfunction and decreased sperm counts (Environment Agency 1998).

1.1 Objective and Scope of Study

The main objective is to apply the upflow anaerobic sludge blanket (UASB) reactor for the treatment of pharmaceutical wastewater. The monitoring parameters will be VSS/TSS, COD, pH, ammonia nitrogen, phosphorus, alkalinity and gas production. The test will cover the raw wastewater and also effluent from (UASB) reactor. Besides, the optimum condition for the anaerobic reactor to remove COD will also be determined.

The outcome of this test will also cover the effect of hydraulic retention time (HRT) and organic loading rate (OLR) on the efficiency of treatment that will be analyzed.

CHAPTER 2

LITERATURE REVIEW

2.0   INTRODUCTION TO PHARMACEUTICAL COMPANY

A pharmaceutical company, or drug company, is a commercial business whose focus is to research, develop, market and/or distribute drugs, most commonly in the context of healthcare. They can deal in generic and/or brand medications. They are subject to a variety of laws and regulations regarding the patenting, testing and marketing of drugs, particularly prescription drugs. From its beginning at the start of the 19th Century, the pharmaceutical industry is now one of the most successful and influential, attracting both praise and controversy. The wastewater for this study is taken from the **Pharmaniaga Group** located in Bangi.

Pharmaniaga Berhad is the pharmaceutical division of the UEM Group, and it is one of the largest integrated local healthcare company in Malaysia. Pharmaniaga is involved in generic pharmaceuticals manufacturing, research and development, housing and distribution of pharmaceuticals and medical products, sales & marketing, hospital equipping and healthcare IT solutions. Some of the registered product from Pharmaniaga Group are InnoHerb Dandelion & Milk Thistle Capsule, InnoHerb Spirulina Capsule, Pharmaniaga Itraconazole capsule 100mg , Pharmaniaga Ofloxacin tablet 100mg and Pharmaniaga Oxymetazoline 0.025% Nasal solution. (http://www.uem.com.my/biz_health.php).

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2.1 Treatment for pharmaceutical effluent

In Ecuador, the treatment that been introduced to treat the pharmaceutical effluent has been treated by using an aeration tank and a secondary clarifier, both of which are in a glass fused to steel bolted tank design as shown in Figure 2.1. This treatment system was installed at the Bristol Myers Squibb factory, the largest medicinal product facilities that serve the South America markets. The system was design for treating wastewater with flow rate of 150 m^3/day with BOD of 500 mg/L and COD of 1000 mg/L. (http://www.uemgroup.com).

In Malaysia, Pharmaniaga Berhad also uses the same treatment and the effluent discharge is reported to comply with Standard A EQA 1974 (refer appendix A1).

Figure 2.1: Combination of aeration tank and secondary clarifier

In Tamil Nadu India, for treating the discharge from the Penicillin-G manufacturing facilities that used sugarcane molasses as a raw material, the combination of Low Rate Anaerobic Digester followed by Aerobic Activated Sludge Treatment System with Fine Bubble Diffusers was introduced. The treatment system supplied by UEM India to treat process wastewater, mycelium and contaminated batches was designed for a flow of 1020 M^3/day, BOD of 9,800 mg/l & COD of 30,880 mg/l can achieve low final BOD levels (<30 mg/l). This system has been working satisfactorily for the

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last several years and is producing the guaranteed results. (http://www.uemgroup.com).

2.2 Anaerobic Wastewater Treatment

Anaerobic is a technical word which literally means without air (where "air" is generally used to mean oxygen), as opposed to aerobic. The rationale for and interest in the use of anaerobic treatment can be explained by considering its advantages that include methane production as a potential energy source, less biological sludge production, and with acclimation most organic compounds can be transformed (Metcalf & Eddy, 2004). Figure 2.2 shows the simplified diagram of anaerobic digestion.

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┌─────────────────────────────────────────────────────────────────────────┐
│  ┌──────────────┐        ┌──────────────────┐      ┌──────────────────┐   │
│  │  ANAEROBIC   │───────▶│     INNOCOUS     │  +   │     BIOGAS       │   │
│  │  DIGESTION   │        │  DIGESTED SOLIDS │      │                  │   │
│  └──────────────┘        └──────────────────┘      └──────────────────┘   │
│                                                              │            │
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│                                                          CH₄+CO₂          │
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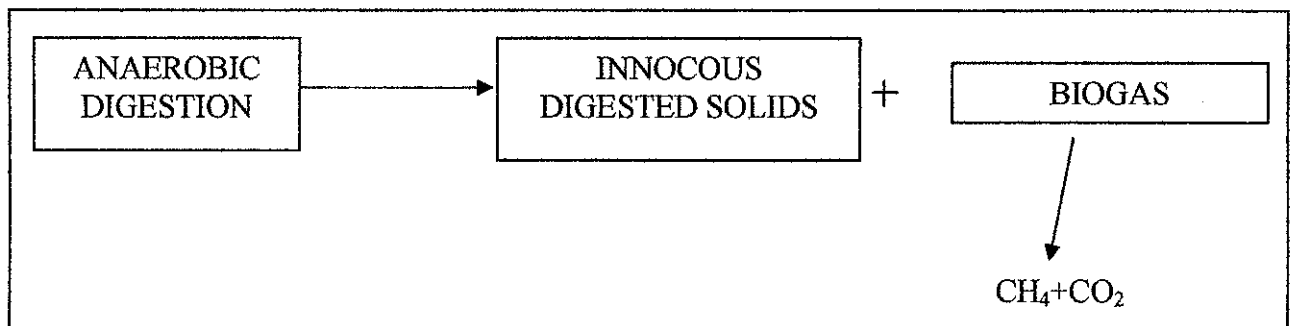
Figure 2.2: Anaerobic digestion

2.2.1 Three- Stages of Anaerobic Conversion of Organic Matter

The first step for most fermentation process, in which particulate material is converted to soluble compounds that can be hydrolyzed further to simple monomers that are used by bacteria for fermentation, is termed hydrolysis (Metcalf & Eddy, 2004).

The second step is fermentation (acidogenesis). In the fermentation process, amino acids, sugars, and some fatty acids are degraded further, as shown in Figure 2.3. Organic substrate serves as both the electron donors and acceptors. The principle

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products of fermentation are acetate, hydrogen, CO_2, and propionate and butyrate. The propionate and butyrate are fermented further to also produce hydrogen, CO_2, and acetate. Thus, the final products of fermentation (acetate, hydrogen, and CO_2) are the precursors of methane formation (methagonesis). The free energy change associated with the conversion of propionate and butyrate to acetate and hydrogen requires that hydrogen be at low concentration in the system ($H_2 < 10^{-4}$ atm), or the reaction will not proceed (McCarty and Smith, 1986).
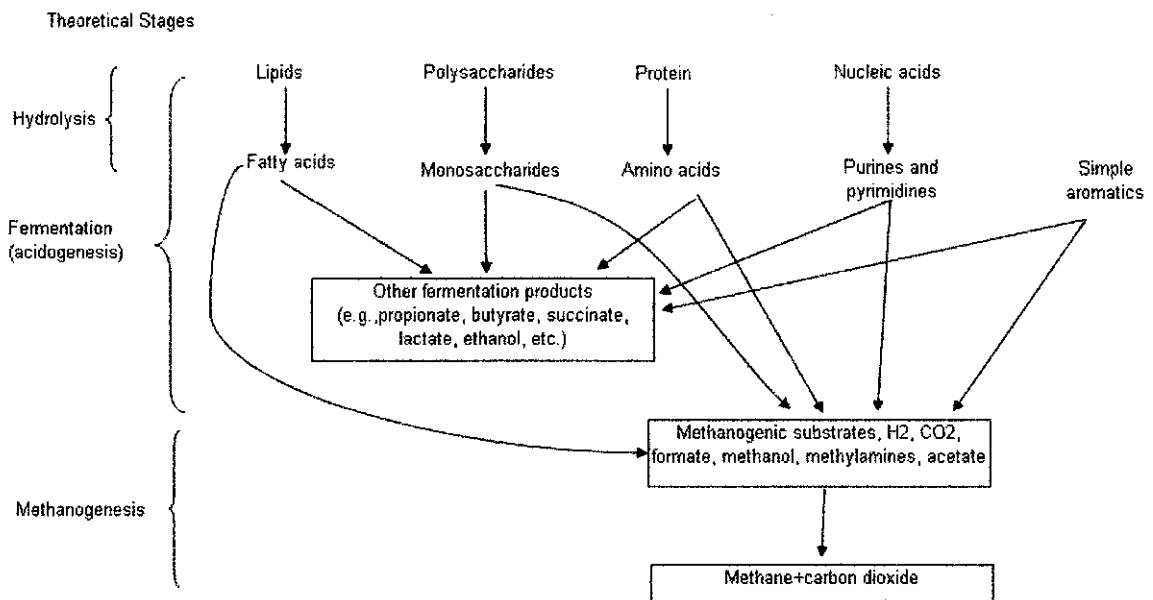
Figure 2.3: Anaerobic process schematic of hydrolysis, fermentation, and methanogenesis. (Adapted from McCarty and Smith, 1991)

The third step, methanogenesis, is carried out by a group of organisms known collectively as methanogens. Two groups of methanogenic organisms are involved in methane production. One group, termed aceticlastic methanogens, split acetate into methane and carbon dioxide. The second group, termed hydrogen-utilizing methanogens, use hydrogen as the electron donor and CO_2 as the electron acceptor to produce methane. Bacteria within anaerobic processes, termed acetogens, are also able to use CO_2 to oxidize hydrogen and form acetic acids. However, the acetic acid will be converted to methane, so the impact of this reaction is minor. As shown in

Figure 2.4 about 72 percent of methane produced in anaerobic digestion is from acetate formation. (Metcalf & Eddy, 2004)
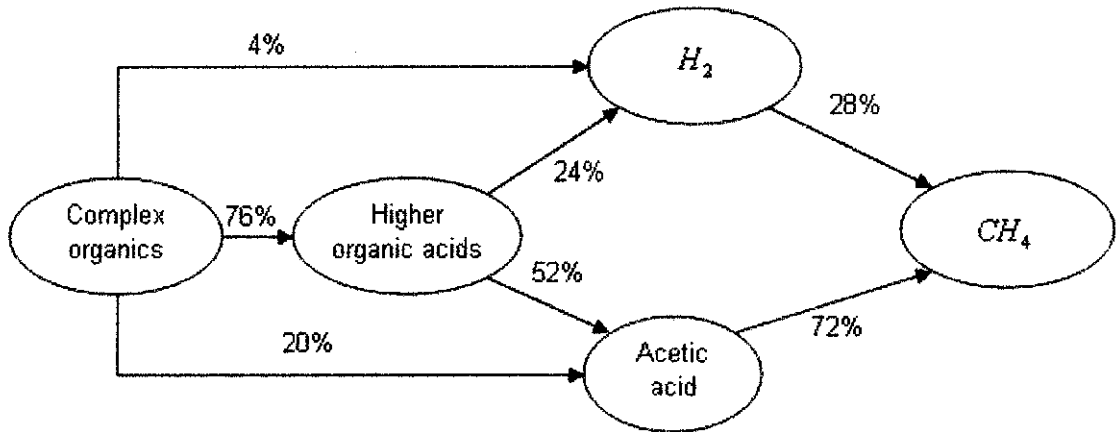
Figure 2.4: Carbon and hydrogen flow in anaerobic digestion process. The given percentage values are based on COD. (Adapted from Jeris and McCarty, 1963 and McCarty, 1981)

The critical biochemical reactions in these stages are presented in Figure 2.5

Hydrolysis

Complex carbohydrates ⟶ Simple sugars

Complex lipids ⟶ Fatty acids

Complex proteins ⟶ Amino acids

Acid Production

Simple sugars + fatty acids + amino acids ⟶ Organic acids, including acetate + alcohols

Acetogenesis (acetate production)

Organic acids + alcohols ⟶ acetate

Methane production : acetoclastic methanogenesis

| Acetate | ⟶ $CH_4 + CO_2$ |

Methane production : hydogenotrophic methanogenesis

| $H_2 + CO_2$ | ⟶ CH_4 |

Methane production : methylrophic methanogenesis

| Methanol | ⟶ $CH_4 + H_2O$ |

Figure 2.5: The critical biochemical reactions in the anaerobic digestion process and production of methane include hydrolysis, acid production, acetogenesis, and methane production. Methane production may occur through the use of acetate, hydrogen and carbon dioxide, and methanol. (The Microbiology of Anaerobic Digester by Gerardi, 2003)

2.3 Types of Anaerobic Reactors

Anaerobic reactors are capable of treating insoluble wastes and soluble wastewater. Based on bacteria growth fashion, these reactors can be classified suspended growth reactor and attached growth reactor.

2.3.1 Suspended growth process

i.   Complete Mix Process

Bacteria are suspended in the reactors through intermittent or continuous mixing. The mixing action distributes the bacteria or biomass throughout the digester. The suspended growth process is divided into several categories stated below. Figure 2.6 shows the diagram of a complete mix reactor under the anaerobic suspended growth process. In this process, the hydraulic retention time is equal to solids retention time. It is suitable for waste that contains high concentration of solids (Metcalf & Eddy, 2004).
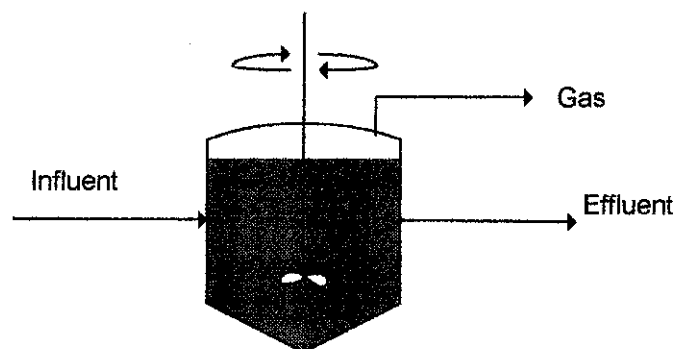
Figure 2.6: Complete mix process diagram

ii. Anaerobic Contact Process

The anaerobic contact process (Figure 2.7) overcomes the disadvantages of complete mix process by having sludge recycle. Gravity separation is the common approach for solids separation and thickening prior to sludge recycle. Sludge with poor settling properties are commonly produced and method to improve solids capture must be employed (Metcalf & Eddy, 2004).
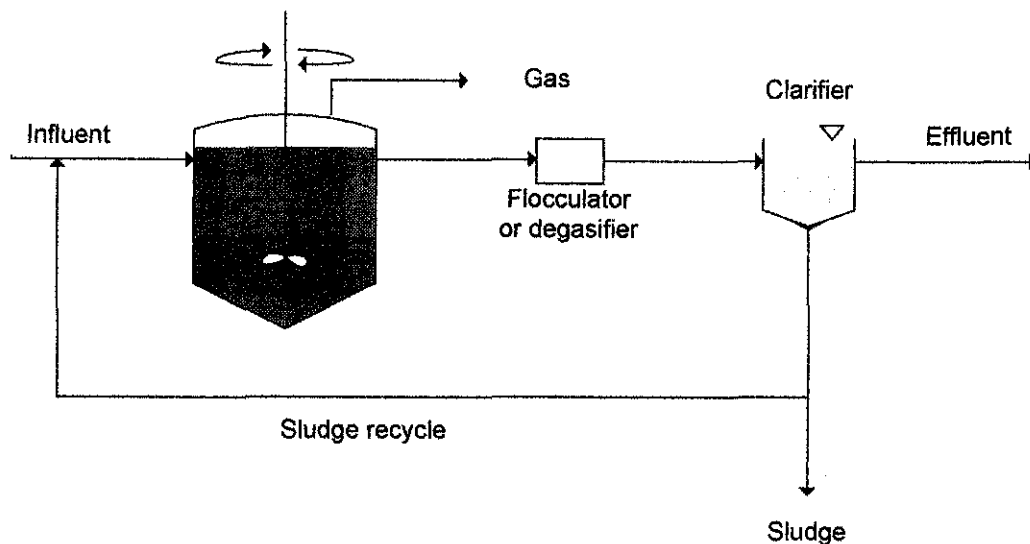
Figure 2.7: Anaerobic contact process diagram

Following are the advantages and disadvantages of suspended growth anaerobic digester.

Advantages	• Suitable for the treatment of particulate, colloidal , and soluble waste
	• Toxic wastes may be diluted
	• Uniform distribution of nutrient, pH, substrate, and temperature
Disadvantages	• Large digester volume required
	• Treatment efficiency may be reduced due to loss of particulate and colloidal wastes and bacteria in digester effluent

2.3.2 Attached growth process

The system provides a quiescent environment for the growth of an agglutinated mass of bacteria. A medium is used to hold bacteria in the digester for relatively long periods and provide long solids retention time and short hydraulic retention time. In the up flow packed bed reactor or anaerobic filter (Figure 2.8), the packing is fixed and the wastewater flow up through the interstitial space between the packing and biogrowth (Metcalf & Eddy, 2004).
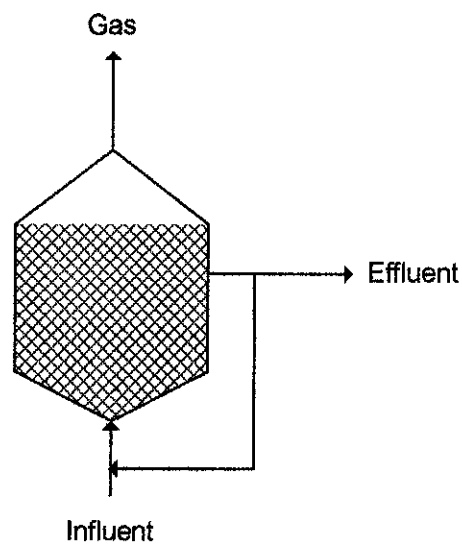
Figure 2.8: Up-flow packed bed reactor or anaerobic filter

Effluent recycle is generally not used for the packed bed reactor except for high-strength wastewater.

2.3.3 UASB (up-flow anaerobic sludge blanket) reactor

The UASB reactor is one of the most notable developments of anaerobic treatment processes that were introduced in the late 1970s in the Netherlands by Lettinga and his coworkers. The UASB process is widely used, with over 500 installations treating a wide range of industrial wastewater worldwide. Basic UASB is illustrated in Figure 2.9. The influent wastewater is distributed at the bottom of the UASB reactor and travels in an up flow mode through the sludge blanket. (Metcalf & Eddy, 2004)
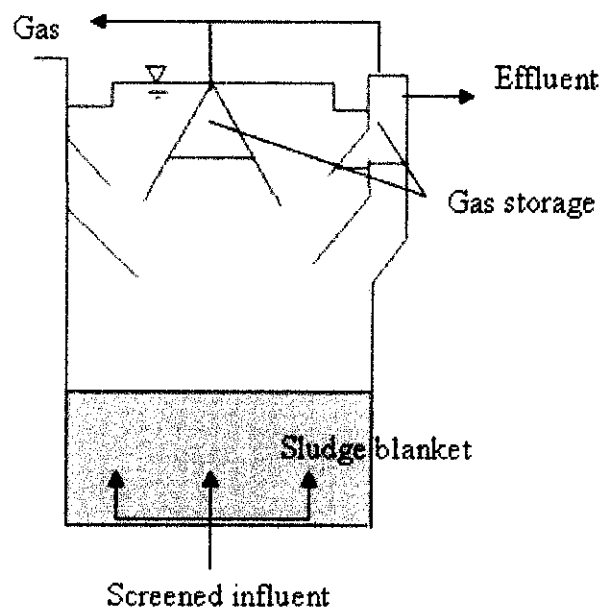
Figure 2.9: Basic UASB Reactor

2.3.4 Methane forming bacteria

Most methane-forming bacteria are active in two temperature ranges, mesophilic range from 30 to 35°C and thermophilic range from 50 to 60°C.

Although methane production can occur over a wide range of temperatures, most of methane forming bacteria are mesophiles. Table 2.1 shows comparison of advantage and disadvantages of mesophilic and thermophilic digesters. The rate of anaerobic digestion is proportional to the temperature, which mean the rate of anaerobic digestion of wastewater and methane production is faster in thermophilic digester compared to mesophilic.

Table 2.1: Comparison of Mesophilic and Thermophilic Digestion

Feature	Mesophilic Digester	Thermophilic Digester
Loading rates	Lower	Higher
Destruction of pathogens	Lower	Higher
Sensitivity to toxicants	Lower	Higher
Operational costs	Lower	Higher
Temperature control	Less difficult	More difficult

(Resource: The Microbiology of Anaerobic Digesters by Gerardi, 2003)

2.4 Temperature

Change in temperature has the most significant impact on the activity of anaerobes and the efficiency of digester performance. Change in temperature also effect the quality and quantity of products obtained through fermentation. These products may or may not be readily available substrates for methane forming bacteria. Therefore, a change in temperature $\pm 2^{\circ}C$ per day should not be permitted and the temperature throughout the digester should be consistent. An acceptable range of temperatures for mesophilic digesters is 30-35°C (Gerardi, 2003).

2.4.1 Mesophilic

Mesophilic digestion and methane production occur at a moderate temperature range (30-35°C). Mesophilic anaerobic digestion is commonly used for municipal and industrial wastewater treatment and offers two practical advantages of operation compared to thermophilic anaerobic digestion. First, there are more anaerobic mesophiles in nature than thermophiles. Second, it is less expensive to maintain mesophilic temperatures in digester than it is to maintain thermophilic temperatures (Gerardi, 2003).

2.4.2 Thermophilic

Thermophilic digestion and methane production occur at a high temperature (50-60°C). Thermophilic anaerobic digestion is more often used at industrial wastewater treatment plants, where process heat or steam is available to heat digesters to thermophilic range. The number of thermophilic methane-forming bacteria is very limited, the bacteria growth is slow, and the bacterial population experiences a high endogenous death rate. Also, the bacteria are very sensitive to fluctuations in digester temperature (Gerardi, 2003).

2.5 Operational Conditions

Methane-forming bacteria are strict anaerobes and are extremely sensitive to changes in alkalinity, pH, and temperature. Therefore, operational conditions in the digester must be periodically monitored and maintained within optimum ranges. In addition to alkalinity, pH, and temperature, several other operational conditions should be monitored and maintained within optimum ranges for acceptable activity of methane forming bacteria. These conditions are gas composition, hydraulic retention times (HRT), oxidation-reduction potential (ORP), and volatile acid concentration (Table 2.2) (Gerardi, 2003).

Table 2.2: Operational Conditions for Acceptable Activity of Methane-forming Bacteria and Methane Production

Condition	Optimum	Marginal
Alkalinity, mg/l as $CaCO_3$	1500-3000	1000-1500
		3000-5000
Gas Composition		
Methane, %volume	65-70	60-65 & 70-75
Carbon dioxide, %volume	30-35	25-30 & 35-40
Hydraulic retention time, days	10-15	7-10 & 15-30
pH	6.8-7.2	6.6-6.8 & 7.2-7.6
Temperature, mesophilic	30-35°C	20-30°C & 35-40°C
Temperature, thermophilic	50-56°C	45-50°C & 57-60°C
Volatile acids, mg/l as acetic acid	50-500	500-2000

(Resource: The Microbiology of Anaerobic Digester by Gerardi, 2003)

2.6 Organic Loading Rate (OLR)

The equation of OLR was determined by liquid volume of reactor equation (Metcalf & Eddy, 2004). The original equation is listed below:

$$V_n = \frac{QS_o}{L_{org}}$$

Where V_n= nominal (effective) liquid volume of reactor

Q = influent flowrate

S_o= Influent COD

L_{org}= organic loading rate

Thus with the know value of liquid volume of reactor the OLR can be determined by substitute the equation below:

$$L_{org} = \frac{QS_o}{V_n}$$

CHAPTER 3

MATERIALS AND METHODS

3.0   METHODOLOGY

Two reactors were used one each operated in mesophilic (30°C-35°C) and thermophilic (50°C -60°C) ranges. Both reactors were monitored simultaneously. The operating temperature for mesophilic reactor was set as 35°C and for thermophilic reactor was set as 54°C. The reactors were operated by using the same influent. Effluent samples for testing were taken from the liquid sampling point (LS). Figure 3.1 shows the schematic diagram of the experimental setup.
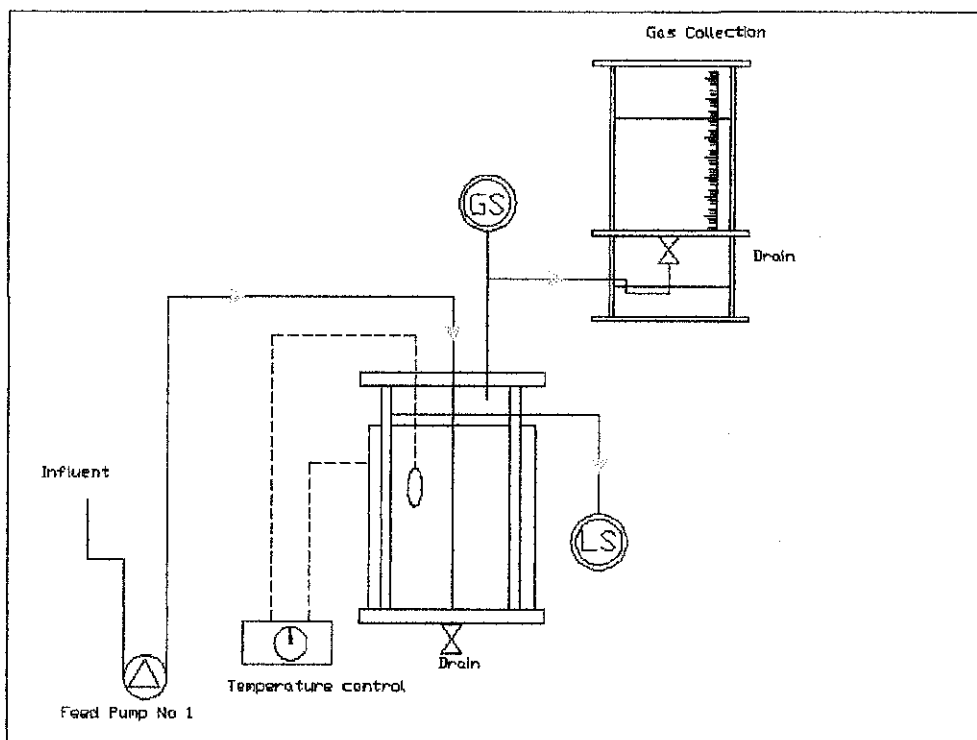
Figure 3.1: Schematic diagram of the experimental setup

The lab work that have was done include the testing for Chemical Oxygen Demand, Total Suspended Solids, Volatile Suspended Solids, Ammonia Nitrogen, pH, Alkalinity and Phosphorus test for influent and both effluent. The peristaltic pumps were calibrated so that the flow rate of the incoming wastewater could be controlled. Those works is done accordingly to the Standard Method.

3.1   Experimental Setup

The upflow anaerobic sludge blanket (UASB) reactors used in this study (Figure 3.2) comprised two 5 litre upward-flow reactors with feed rate and temperature control facilities to allow steady, continuous operation at up to seven litres per day. The reactors were operated in parallel.

The temperature of each reactor was controlled by an electric heating mat wrapped around the external wall. The temperature distribution within each reactor was maintained at ±1°C of the desired temperature.

The gas off-take from each reactor was taken to a volumetrically calibrated collector vessel operating by water displacement. A constant head, liquid seal device ensured that the gas pressure in the reactor was maintained at a constant value throughout the test run. The collected gas can be exhausted from the vessel and the volume re-filled with water during a run without breaking the liquid seal.

Liquid and gas sampling points were located at all strategic points around the reactors. Non-return valves and liquid seal siphon breaks were included in the process pipe work to ensure each reactor operated at a constant volume without the ingress of air or the danger of accidental syphonic action.

The equipment was mounted on a vacuum formed plastic base with an integral drain channel to cope with spillages and wash down. (Armfield Catalogue, issue 4)
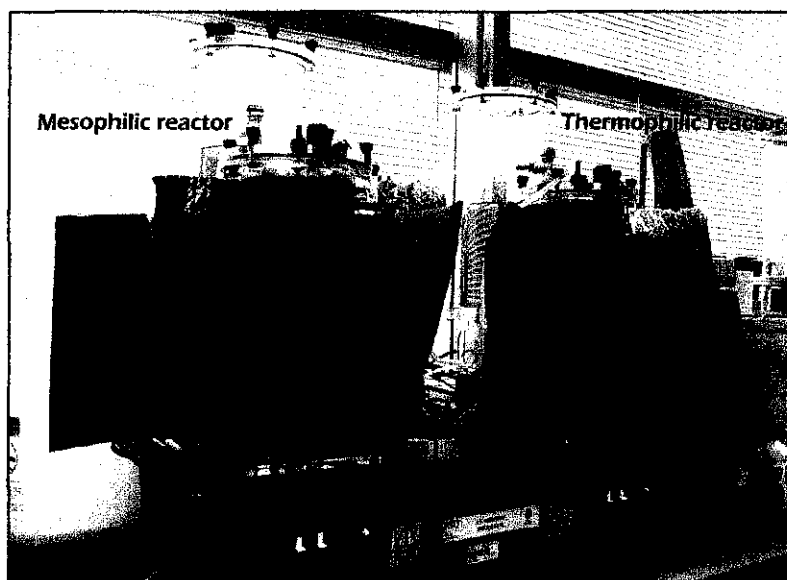
Figure 3.2: Up-flow Anaerobic Sludge Blanket Reactor

3.1.1 Technical Details of Anaerobic Reactor (UASB)

Reactors   : 2 reactors, identical

Nominal capacity   : 5 litres

Dimension   : 150 mm diameter x 250mm high

Reactor packing   : no packing

Temperature control   : 200W heating jacket for each reactor

Feed pumps   : pneumatic pump, 10 turn potentiometer to 4rpm

Flow rate   : 0.2 – 5.8 L/day

Gas collection vessels : 2 identical vessel, 0-5 litre capacity

3.2　Reactor Startup and Monitoring

The UASB reactors were seeded with sludge taken from the sludge thickener at campus wastewater treatment plant (STP) located at Universiti Teknologi PETRONAS (UTP). The hydraulic retention time was initially set at 5 days. Table 2 provides the test schedule for the study period. Test procedure generally followed standard methods for waste and wastewater treatment (APHA, 1995). The monitoring and sampling of the influent and both effluent were done routinely as show in Table 2. In one week, there will be at least 3 test conducted.

Table 3.1: Monitoring frequency

Test	Desired Monitoring Frequency	
	Routine	As required
Alkalinity	√	
Chemical oxygen demand (COD)	√	
Gas composition	√	
Gas production	√	
Ammonia Nitrogen	√	
Total phosphorus	√	
pH	√	
VSS/TSS		√

3.3   Flow rate measurement

The flow rate is controlled by the peristaltic pump (Figure 3.3). The preliminary work had been done to obtain the suitable flow rate. The pump will control the flow rate of the incoming raw waste water that would go in both reactors.
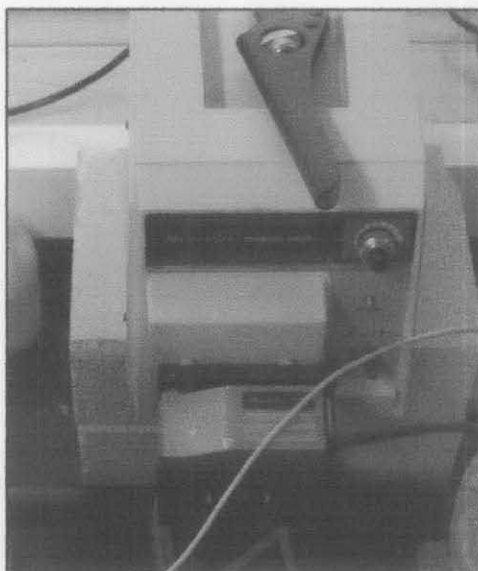
Figure 3.3: Peristaltic pump

Figure 3.4 shows the calibration graph for the pump. The time was taken for the water to fill up certain volume. Based on the volume and time, the flow rate was obtained as shown in equation below:

$$Q = \frac{V}{t}$$

Where:

Q = flow rate

V = volume

t = time
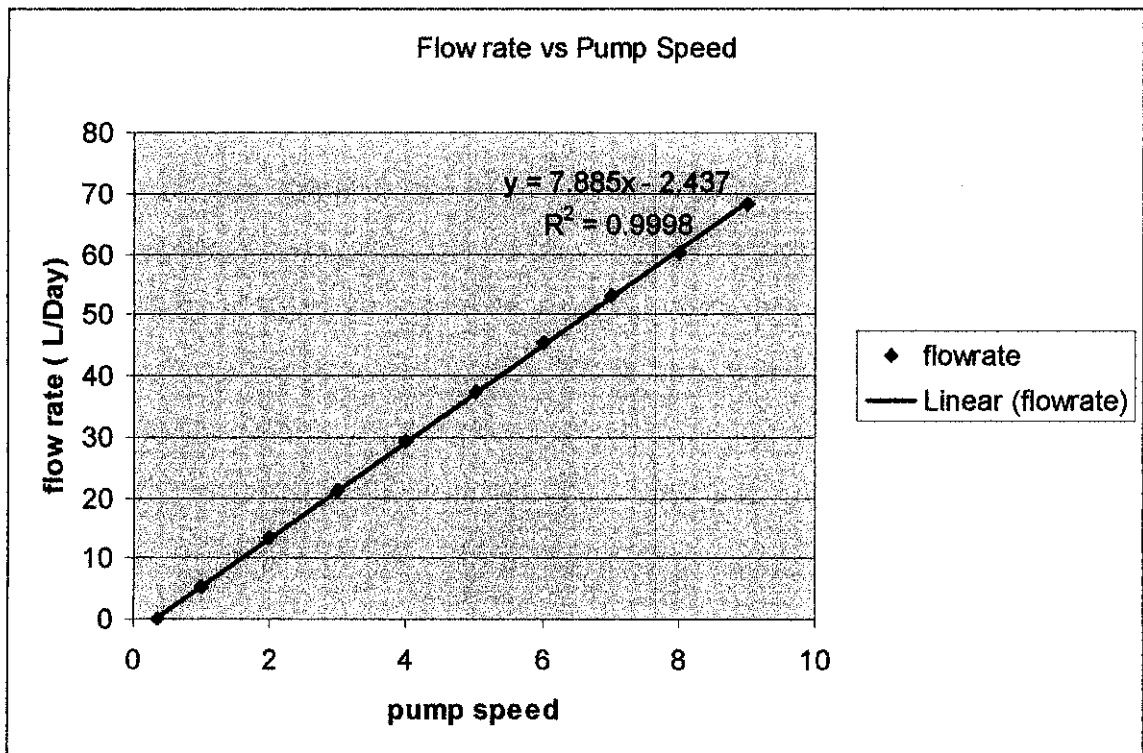
Figure 3.4: Graph of flow rate against pump speed

Thus by controlling the flowrate, the Hydraulic Retention Time (HRT) can be controlled. The HRT was obtained by as shown in equation below:

$$HRT = \frac{V}{Q}$$

Where

V = volume of reactor

Q = flow rate

Since the reactor volume is 5 liter, the correlation between HRT and flowrate was shown in table 3.2

Table 3.2: Pump calibration table

HRT, (day)	Flowrate Q, (Liter/Day)	Pump Speed
1	5	0.943
2	2.5	0.626
3	1.67	0.52
4	1.25	0.468
5	1	0.435

Table 3.2 will be used as reference when there is need to change the HRT of the reactors.

3.4   Reactor seeding

The reactor was seeded with the sludge obtained from the sludge thickener located at the Sewage Treatment Plant (STP) at Universiti Teknologi PETRONAS. 2.5litre sludge with 16277 mg/L MLVSS was poured inside the reactor. Then the pharmaceutical wastewater was pour until the volume was 5 liter.

3.5 Gas production

The gas production was monitored routinely by reading the gauge located at the collector vessel. As shown in Figure 3.5, 5% Sodium Hydroxide solution with Themol Blue indicator was used as displacement liquid (Isa, et al., 1993). The reading of the gas level was guided by the level indicator at the gas vessel. Due to the absorption of Carbon Dioxide by the Sodium Hydroxide, the gas collected will be expected as pure Methane (CH_4).
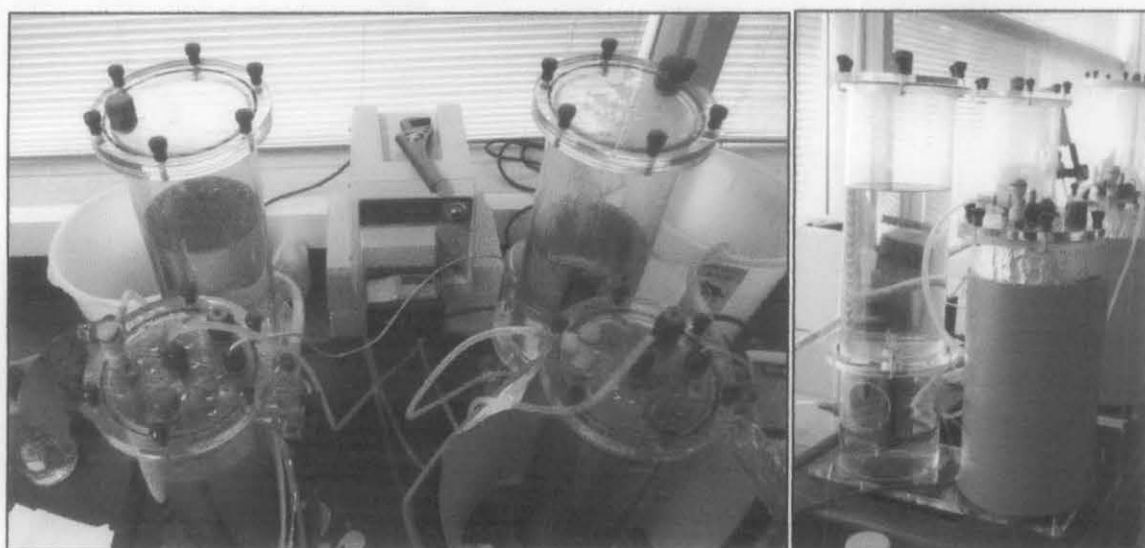
Figure 3.5: Topside view and side view of reactor with the gas vessel.

3.6 Total Suspended Solids

TSS was determined using Whatman® Filter Papers Cat No 1 with diameter of 47 mm pore size ranging (0.45μm to about 2 μm). It is also important to note that the TSS itself has no fundamental significance. The test of TSS will be conducted for influent of waste water and also both effluent. The aluminum foil and the filter paper must first be heated for 105°C for an hour to remove all moisture that would affect the weight reading later on. Figure 3.6 shows test apparatus involved in the Total Suspended Solids test.

Figure 3.6: TSS and VSS test apparatus.

3.7 Volatile Suspended Solids

Material that can be volatilized and burned off when ignited at 550°C is classified as volatile. In general, volatile solids (VS) are presumed to be organic matter. In the lab works, after the work done for TSS, the VSS test served as the continuation of the work. The sample was inserted in a furnace (Figure 3.7) ignited at 550°C. During this period, the filtered organic matter will be burned. The sample left in the foil will be called Fixed Solids and the sample that evaporated will be referred as the Volatile solids. Results obtained were reported as VSS/TSS.

Figure 3.7: The furnace with the ignition of 550°C

3.8 Chemical Oxygen Demand (COD)

The COD test is used to measure the oxygen equivalent of the material in wastewater that can be oxidized chemically using dichromate in acid solution. From an operational standpoint, one of the main advantages of the COD test is that it can be completed in about 2.5 h, compared to 5 or more days for BOD test. During COD test, the apparatus that were being used was the digestion solution for COD 0-1500 ppm by HACH. The process involved the usage of the DRB 200 Heater by HACH and DRB 2800 Spectrophotometer from the HACH company (Figure 3.8).

Figure 3.8: COD test apparatus

3.9 Ammonia nitrogen

The nitrogen is essential as requirement for the nutrient need from the anaerobic digestion process. The method used according to the finding of the ammonia nitrogen was by the adaptation of Nessler method. The importance of the test was to monitor the nutrient requirement for the anaerobic digestion. Proper dilution need to be done before conducting the test to avoid reading over ranged. The procedure for doing the ammonia nitrogen test involved the mixing of three solutions which were Mineral Stabilizer, Polyvinyl Alcohol and Nessler Reagent (Figure 3.9).

Figure 3.9: Ammonia Nitrogen by using Nessler Method apparatus

3.10 Total phosphorus

Total phosphorus test plays the same important rule such as the ammonia nitrogen test. Before conducting both tests, proper dilution was needed because the sample reading without any dilution will be higher than the spectrometer reading range. The test will be conducted by using Total Phosphate Test 'N Tube Reagent set 0-3.5 mg/L PO_4^{3-} by HACH as shown in Figure 3.10. The test involved the DRB 200 Heater and the reading for the total phosphorus concentration will be taken from the DRB 2800 Spectrophotometer. The test involved the mixing of the Pottasium Persulfate Powder, Sodium Hydroxide and Phos Ver 3 powder. Precaution must be taken when opening the Phos Ver 3 powder since some of the powder pack was contaminated known by the brownish color of the powder. The powder was originally white in color.

3.10: Total Phosphorus test apparatus

3.11 Alkalinity and pH

Sufficient alkalinity is essential for proper pH control. Alkalinity serves as buffer that prevents rapid change in pH. Enzymatic activity or digester performance is influenced by pH. The pH of the influent and effluent of were measured using the Mettler Toledo MP230 pH meter as shown in Figure 3.11. Sometimes the usage of the Sension 4 PH ISE meter by HACH will be used if there were no availability of the first pH meter.
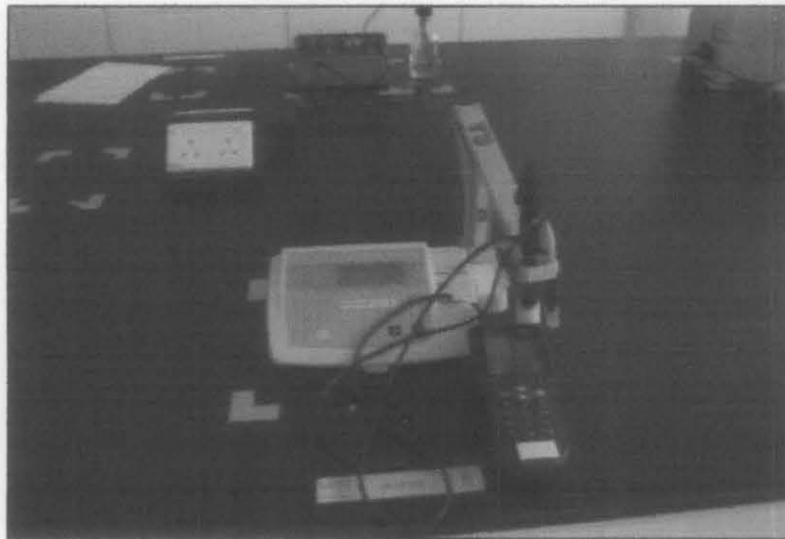
Figure 3.11: pH meter

All forms of alkalinity (hydroxide, carbonate and bicarbonate) can be measured by titration with a standard solution of an acid (0.02N H_2SO_4) and represented in terms of equivalent $CaCO_3$/L. For samples with initial pH above 8.3, the titration is made in two stages. First titration is carried out until the pH is lowered to 8.3 (phenolphthalein end point). This is followed by titration of the aliquot to pH 4.3 (methyl orange end point). When the pH of sample is less than 8.3, single titration to pH 4.5 is made (UTP Environmental Lab Manual).

3.12 Hazard Analysis

For the safe counter measure for doing the lab work. Job safety analysis (JSA) was prepared for the whole lab work (Appendix D). By following the safety measurement, accident can be avoided thus reduce incident that might happen to the personnel during the lab work session. The importance of JSA besides ensuring safety is for the lab work to go on smoothly as personnel can detect any mislead action concurrent to the procedure.

3.13 Project Milestone

The lab works for Final Year Project 2 started in December and finished in mid of April; the schedule work started on 24th December and was expected to finish on 11th April (Appendix C). However, due to some problem encountered which included reactor operation disruption during the Final Year Project period, the lab works have been done at the end of April. The lab works cover the experiments from the COD test until the methane production.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Results

Upon arrival of the wastewater from the Pharmaniaga Group company, several tests were conducted to determine the characteristics of the pharmaceutical wastewater. Table 4.1 concludes the result of the findings. The wastewater was divided into two different strength based on the COD concentration; the first phase until the second phase was the low strength wastewater ranging from 300-500 mg/L while during the third phase until the fifth phase the wastewater characteristic changed to the High Strength wastewater with COD concentration ranging from 1700-2000 mg/L. The first phase covers the preliminary work done during the Final Year Project 1, while for the second until the fifth phase covers the final laboratory work during the Final Year project 2.

Table 4.1: Pharmaceutical wastewater characteristics

Phase	pH	TSS (mg/L)	COD (mg/L)	Phosphorus (mg/L)	Ammonia Nitrogen (mg/L)
1-2	4.47	45	300-500	6-10	n/a
3-5	4.45	32	1700-2000	10-30	3-18

Two anaerobic reactors each operating at different temperatures (mesophilic and thermophilic) were monitored for a total of 221 days. Initially the influent COD of wastewater was in the range of 300-500mg/L at OLR 0.1 kg COD/m^{3}d with hydraulic retention time (HRT) of 5 days. After day 127, the influent COD increased to near

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2000 mg/L because the wastewater characteristic that have been change, causing the organic loading rates (OLR) to increase up to 0.45 kg COD/m^3d and the HRT was increase to 5 days. Summary of the reactor performance was concluded in Table 4.2

Table 4.2: Performance of anaerobic reactors

phase	Day	HRT (d)	OLR (kg COD /m^3d	CH$_4$ production		percentage COD removal %	
				M	T	M	T
1	1-17	5	0.07-0.11			70-85	50-75
2	94-127	3	0.11-0.16	150-300	50-200	75-90	60-85
3	135-166	5	0.35-0.45	150-280	90-150	85-95	80-93
4	167-200	4	0.45-0.50	50-250	110-130	35-60	80-85
5	200-211	3	0.50-0.56	80-160	32-176	65-70	75-80

M= mesophilic reactor

T= thermophilic reactor

4.2   COD reduction

Samples were taken routinely from the influent feed tank and also from both reactor effluents, i.e. mesophilic reactor and thermophilic reactor. Since the concentration of pharmaceutical wastewater changed on day 135, the study was divided to five separated phases operating at different Organic Loading Rates (OLR) and Hydraulic Retention Times (HRT).

4.2.1   Phase 1

Phase 1 consisted of the preliminary work done during the Final Year Project 1. The test that had been carried out was the COD test. COD measurement was started after 4 weeks of acclimatization period. The COD concentration in the influent was in the range of 350-500 mg/L. The concentration of effluent in mesophilic and thermophilic reactors was in the range of 70-100 mg/L and 90-170 mg/L. Based on Figure 4.1, the performance of thermophilic reactor was not as good as the percentage of COD

removal was within the range of 50%-75% as compared to mesophilic reactor which was 70%-85%. The organic loading rate for the Phase 1 was in the range of 0.07-0.11 kg COD/m^3d. The hydraulic retention time (HRT) for Phase 1 was 5 days.

4.2.2   Phase 2

Phase 2 started on day 94 and continued up to day 127. The flowrate was increased causing the hydraulic retention time to decrease from 5 days to 3 days. During Phase 2, the organic loading rate was in the range of 0.1 kg COD/m^3d to 0.16 kg COD/m^3d (Figure 4.2).The COD concentration of the influent ranged about 300-400 mg/L. The final COD concentration for mesophilic reactors effluent was 50-80 mg/L while for the thermophilic reactor, the concentration was 70-115 mg/L (Figure 4.1). For the effluent from the mesophilic reactor, the discharge quality in term of COD met the standard B limits of EQA 1979 throughout (refer Appendix A). The percentage of COD removal for the mesophilic reactor was in the range of 75%-90% while for thermophilic reactor the percentage COD removal was in the range of 60%-85%. Phase 1 to Phase 2 was classified as low strength of wastewater due to lower influent COD concentration compared to other phases.
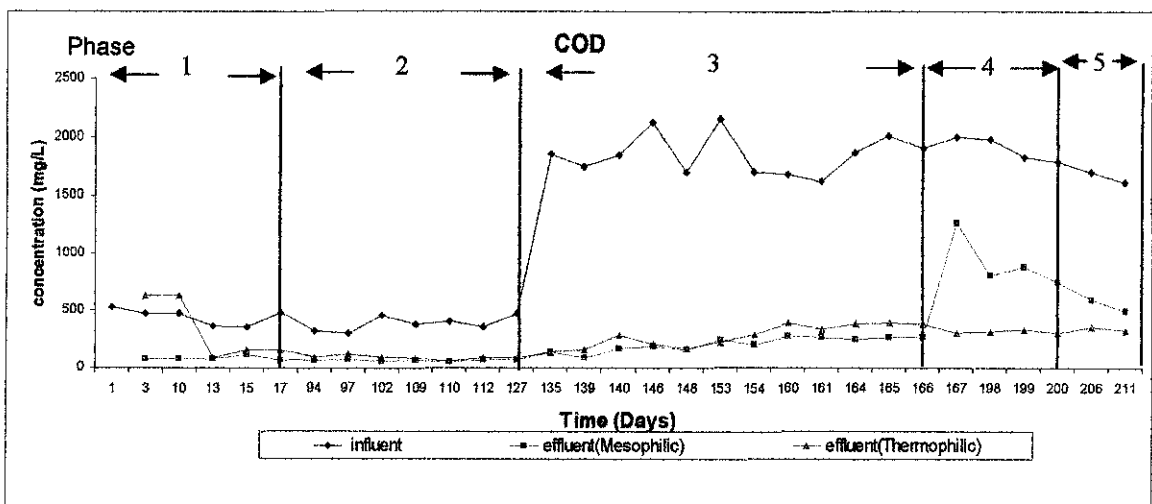

Figure 4.1: COD reading of anaerobic reactors

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4.2.2 Phase 3

During Phase 3 (135-166 days) the influent COD increased to near 2000mg/L, the organic loading rate also increased due to increase of the influent COD. The organic loading rate was increased in the range of 0.35 kg COD/m³d to 0.45 kg COD/m³d with HRT of 5 days. Both mesophilic and thermophilic reactors have high percentage of COD removal up to 90 percent. For the effluent of mesophilic reactor, the COD concentration ranged from 150 mg/L to 270 mg/L. For the thermophilic reactor, the effluent concentration was quite high compared to mesophilic reactor effluent where the reading ranged from 280 mg/L to 380 mg/L. The COD percentage removal for mesophilic reactor was 85%-95% while for the thermophilic reactor was 80%-93%.

4.2.3 Phase 4

Phase 4 operation was from day 167-200. it was realized later that in the beginning of this phase, the thermostat of the mesophilic reactor broke, causing an increase in reactor content temperature to over 60°C. This resulted in disruption of smooth operation of the mesophilic reactor due to temperature shock to mesophilic microorganism. The effluent become brown in colour due to sludge wash out. COD concentration for mesophilic effluent became high (over 1000 mg/L). The reactor was shut down for two weeks for mitigation. The lab work was continued after the repair work was done. When the reactor was restarted, the effluent concentration of the mesophilic reactor was higher compared to the thermophilic effluent. The COD concentration of mesophilic reactor was in the range of 800 mg/L to 1200 mg/L compared to thermophilic reactor which were 300 mg/L to 330 mg/L. The removal efficiency of mesophilic reactor also decrease with the highest value being only 60 percent. The percentage of COD removal for the thermophilic reactor was in the range of 80-85%. The organic loading rate is increase to 0.5 kg COD/m³d (Figure 4.2). The hydraulic retention time was decreased from 5 days to 4 days.

4.2.4 Phase 5

During phase 5 (day 200-211), the flowrate was increased causing the hydraulic retention time to reduce to 3 days. This caused the OLR to increase up to 0.56 kg

COD/m³d (Figure 4.2). The Mesophilic effluent concentration was in the range of 490 mg/L to 600 mg/L compared to thermophilic reactor effluent which was 320 mg/L to 360 mg/L. The COD percentage removal for mesophilc reactor was in the range of 65-70% while for the thermophilic reactor, the percentage of COD removal was 75-80%.
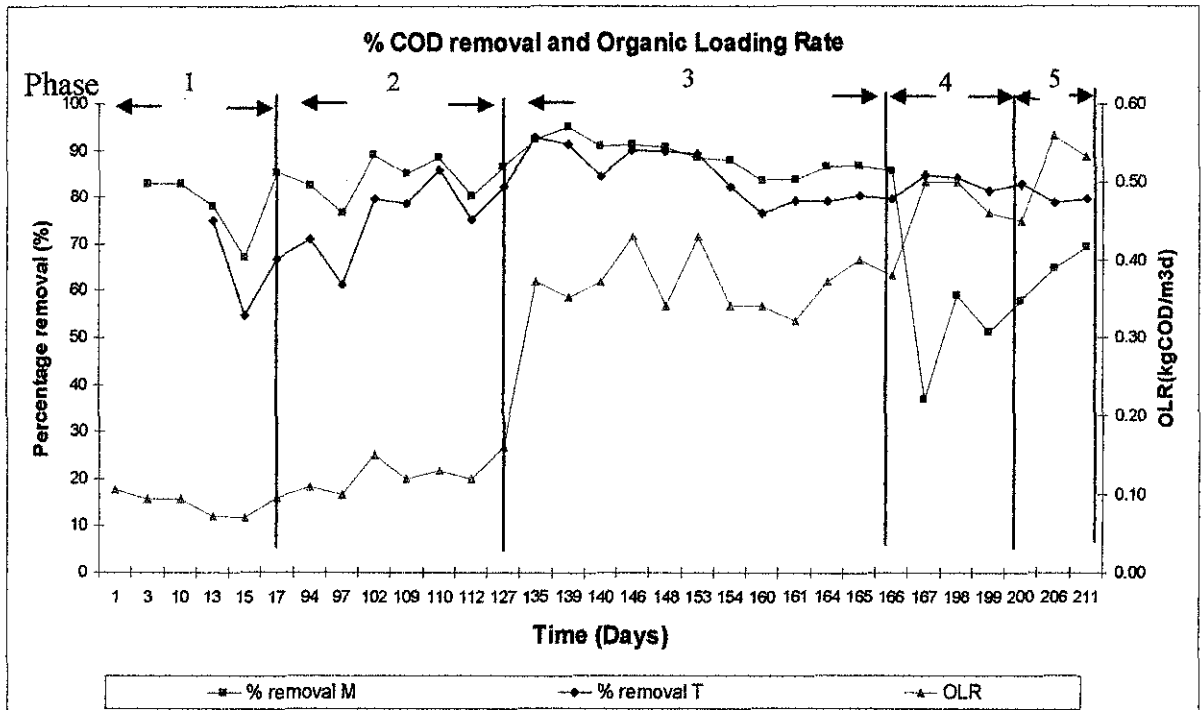
Figure 4.2: Percentage removal of COD with correlation of OLR

Phase 3 has the highest percentage removal of COD with OLR in the range of 0.35 kg COD/m³d to 0.45 kg COD/m³d with HRT of 5 days and both reactors performing at COD removal near 95%. Mesophilic offer higher percentage of COD removal compared to thermophilic reactor from Phase 1 to 3 but during Phase 4 the thermophilic reactor performed better on COD removal as the mesophilic reactor failed to operate efficiently due to temperature shock to microbes.

The stirring processes have to be done to make sure that the wastewater mix properly to avoid fluctuation on the influent COD result. During the phase change from Phase 2 to Phase 3, the result of COD in mesophilic reactor and thermophilic reactor was quite low ranging from 100 mg/L to 200 mg/L (Appendix E), this happened because the flushing out of the remaining wastewater from the previous phase. Since the

flowrate of the pump was decrease to 1 Liter/day to make sure the hydraulic retention time to be maintained at 5 days, the time taken for reactor to achieve stabile state would be more than 5 days. At day 146, both reactors became stable in term of the effluent COD. Increase of the Organic Loading Rate (OLR) caused both reactors to work efficiently at removal up to 90 percent (Figure 4.2).

Higher organic loading rate may reduce the performance of the anaerobic reactor. By referring to Figure 4.2 for overall performance, the increment of the organic loading rate will cause the reactor to perform at slightly lower efficiency due to shorter HRT causing lesser removal of COD as compared to longer HRT for the same influent concentration.

4.3   Methane production

Methane production measurement was started during the final year project 2 periods starting from day 94 to day 211. During Phase 2 with the range of 75 – 90 % of COD removal, the methane production was in the range of 150-300 mL in the mesophilic reactor while with 60-85% COD removal, the methane production was in the range of 50-200 mL in thermophilic reactor. For Phase 3, the methane production was at 150-280 mL and 90-150 mL for mesophilic and thermophilic reactors respectively. During Phase 4, 50-250 mL and 110-130 mL of methane production occurred in the mesophilic and thermophilic reactors respectively. During the final phase or Phase 5, the methane production was 80-160 mL and 32-176 mL for mesophilic and thermophilic reactors. Contrary to expectation, this study did not find a significant relationship between percentage of COD removal and methane production. Based on Figure 4.3, the highest rate of methane production occurs at phase 3 with organic loading rate in the range of 0.35 kg COD/m^3d to 0.45 kg COD/m^3d with HRT of 5 days.
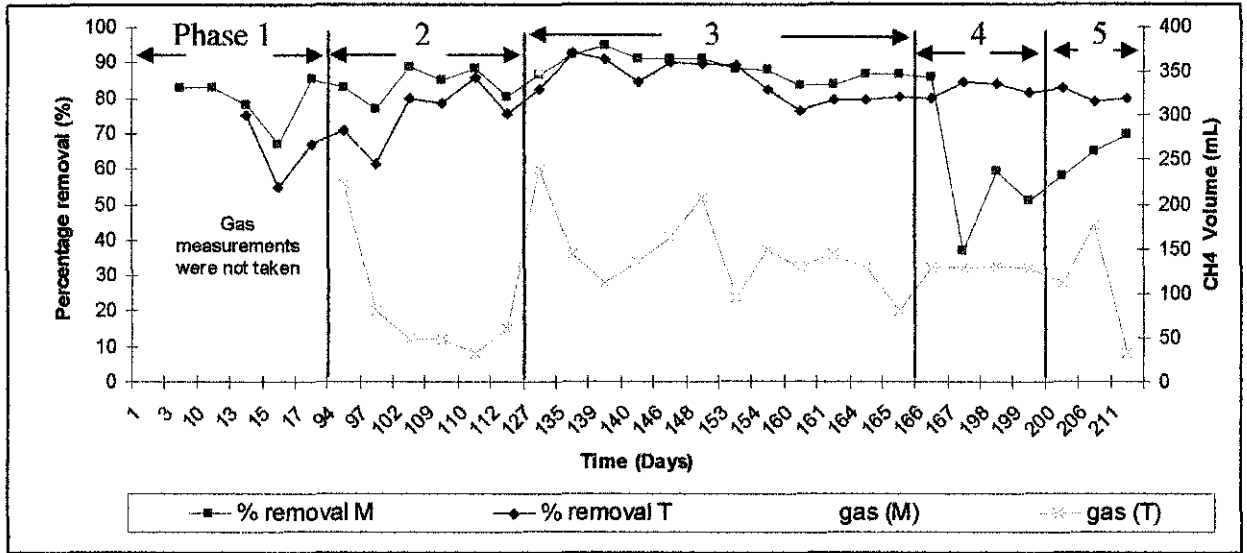
Figure 4.3: COD removal and Methane production

The result (Figure 4.3) hadn't shown any significant improvement for the methane production even though the removal of COD was increased to almost 1500 mg/L. The theoretical production of methane was supposed to be more than 350 mL, compared to the actual data which was less than 300 mL (Figure 4.4).
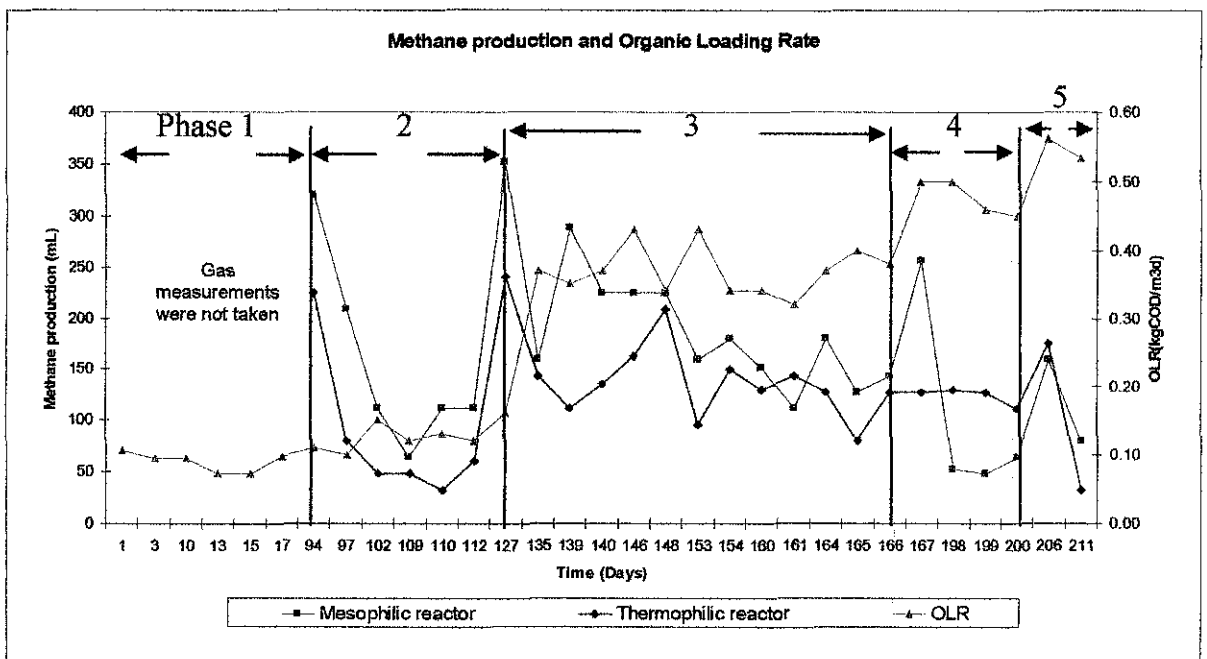

Figure 4.4: Methane production and OLR

The reason for this is not clear but it may have something to do with the instability of temperature and pH. As noted in N.F Gray, 2004 pg 770, the acetogenic and methanogenic bacteria are particularly sensitive to the temperature, with even 2-3°C drop in mesophilic digester will adversely affecting to the biogas production. Even though the allowable temperature changes during the lab work is in range of 1°C. There maybe times when the temperature of the reactors increase more than allowable range and it can be conclude in as reactors inconsistency to maintain temperature. This caused the methane production to be low when compared to the theoretical value. The leakage that may occur at gas collector vessel and also at tube seems to be main reason that contributes to lower methane production. The pharmaceutical wastewater itself may content substance that may cause the production of methane to be low. It can also be assume that the methanogenic bacteria failed to work due to lower alkalinity at the effluent (Figure 4.7).

4.4   Ammonia Nitrogen

Ammonia nitrogen test started at in Phase 3. In this Phase 3, ammonia nitrogen concentration of influent was 2-9 mg/L while at the effluent for mesophilic and thermophilic reactor the ammonia nitrogen concentration ranged from 5-12 mg/L. For Phase 4, the ammonia nitrogen concentration for the influent was in the range of 5-13 mg/L while for mesophilic reactor in the range of 17-32 mg/L and 9-13 mg/L for thermophilic reactor. For the final phase, the ammonia nitrogen concentration for influent was of 7-18 mg/L and 6-14 mg/L for both mesophilic and thermophilic reactors. Ammonia nitrogen concentration at the effluent is higher compared to the influent. During phase 4, it can be seen that the ammonia nitrogen concentration of the mesophilic reactor was relatively high. This is due to reactor instability because of the failure incident. During day 139 and day 148, the ammonia nitrogen in influent is higher than effluent from the thermophilic reactor. This condition is not acceptable and the error may happen because the dilution done for the influent was done in the contaminated flask causing unacceptable range of result occurred. The ammonia ions produced in the reactor as the results of bacterial degradation of amino acids and proteins. Thus the concentration of ammonia nitrogen at the effluent is higher than the influent.

Ammonia nitrogen is essential for anaerobic digestion but can be inhibitory when present at concentration of >150 mg/L. The toxicity threshold for ammonia has been reported to be 100mg/L as NH_3-N (McCarty and McKinney, 1961). Based on Figure 4.5, it can be concluded that the ammonia nitrogen concentration at mesophilic reactor, is higher than the concentration of ammonia in the thermophilic reactor and in the influent.
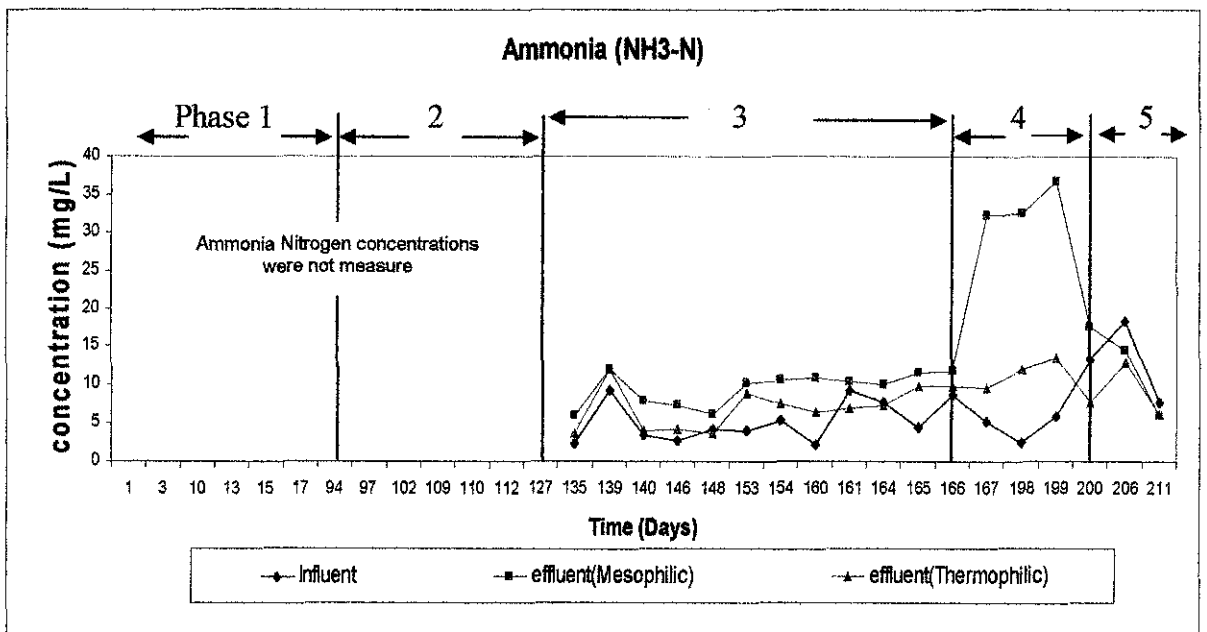
Figure 4.5: Ammonia Nitrogen graph versus time

4.5   Total phosphorus

During Phase 2, the concentration of phosphorus in mesophilic reactor effluent ranged from 9-40 mg/L while for thermophilic reactor the total phosphorus concentration was in the range of 11-38 mg/L. The influent concentration was lower than the effluent with the range being 4-10 mg/L. During Phase 3, the influent concentration is it the range of 10-25 mg/L while for the mesophilic and thermophilic effluent, the concentration ranging in 15-30 mg/L and 16-40 mg/L. In the Phase 4, the effluent of mesophilic reactor is high because of reactor instability and the concentration high may due to sludge washout. The concentration was in the range of 40-80 mg/L and 18-19 mg/L for mesophilic and thermophilic reactors. The influent concentration for Phase 4 was in the range of 12-15 mg/L. During the final phase, the influent

concentration in the range of 2-14 mg/L and for the mesophilic and thermophilic effluent, the concentration was in the range of 30-32 mg/L and 20-22 mg/L.

During Phase 2 and Phase 3, the thermophilic reactor produced higher phosphorus compared to mesophilic reactor. While during the Phase 4 and 5, reactor instability cause the phosphorus concentration of mesophilic increased.

Gerardi (2003) indicated that nutrient requirement vary greatly at different organic rates. Generally, COD: N: P of 1000:7:1 and 350:7:1 have been used for high-strength wastes and low loadings. Based on Figure 4.5 phosphorus in the influent is high (>2 mg/L) and more than required. However, the findings of the current study do not support the previous research when the concentration of effluent is not reduced instead in increase at ranger higher than the influent. This is because bacteria have the capability of storing excess amount of phosphorus as polyphosphates in their cells. Under anaerobic condition, phosphorus accumulating organism (PAOs) will assimilate fermentation products into storage product within the cell with concomitant release of phosphorus from stored polyphosphates (Metcalf & Eddy, 2004). Thus as shown in Figure 4.6, both effluents have higher concentration compared to the influent.
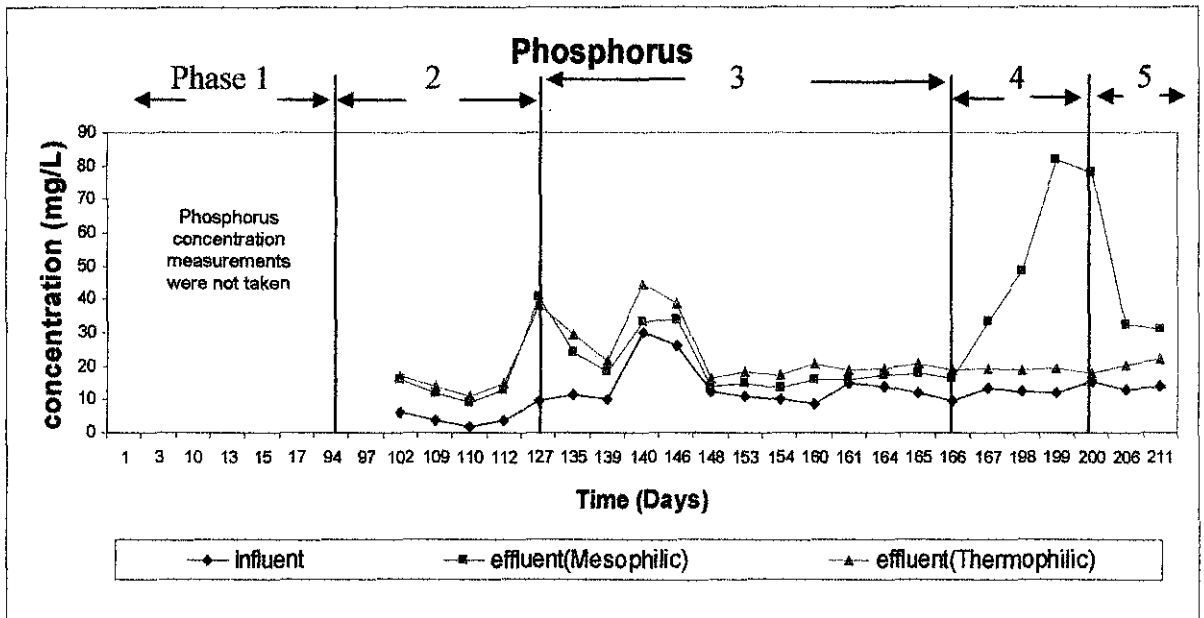
Figure 4.6: Phosphorus concentration graph versus time (days)

4.6 Alkalinity and pH

Figure 4.6 present the alkalinity concentration through out the experiment. During phase 2, the influent alkalinity was in the range of 500 to 950 while the mesophilic and thermophilic effluent were ranging from 500 to 1080. During phase 3, the alkalinity for the influent was not adjusted at first. This caused the pH of the influent (Figure 4.7) to drop to near 4. The pH was then adjusted by adding sodium bicarbonate at 4 gram per 1 liter to boost the alkalinity to near 2000 and achieve pH of 7 at the influent. During Phase 3, the influent alkalinity was in the range of 1500-2080 while for the mesophilic and thermophilic effluent, the alkalinity was in the range of 1000-2250 and 1000-1900. From Figure 4.6 the influent alkalinity is higher compared to both effluents because at least 5 days required for the adjusted alkalinity of the influent to flow out from the effluent. During Phases 4 to 5, the alkalinity for the effluent was higher compared to both effluents. This condition was not accepted because the degradation of amino acids and protein in the anaerobic reactors will cause higher alkalinity at the effluent (Gerardi, 2003). Generally, in anaerobic treatment decrease in the alkalinity may be caused by:

1) accumulation of organic acids due to failure of methane-forming bacteria to convert the organic acids to methane

2) slug discharge of organic acids to the anaerobic reactor

3) presence of waste that inhibit the activity of methane-forming bacteria

High alkalinity is required for the proper control of pH inside reactor. The anaerobic digestion will produce biogas that contains carbon dioxide. High concentration of carbon dioxide can alter the pH inside the anaerobic reactors.
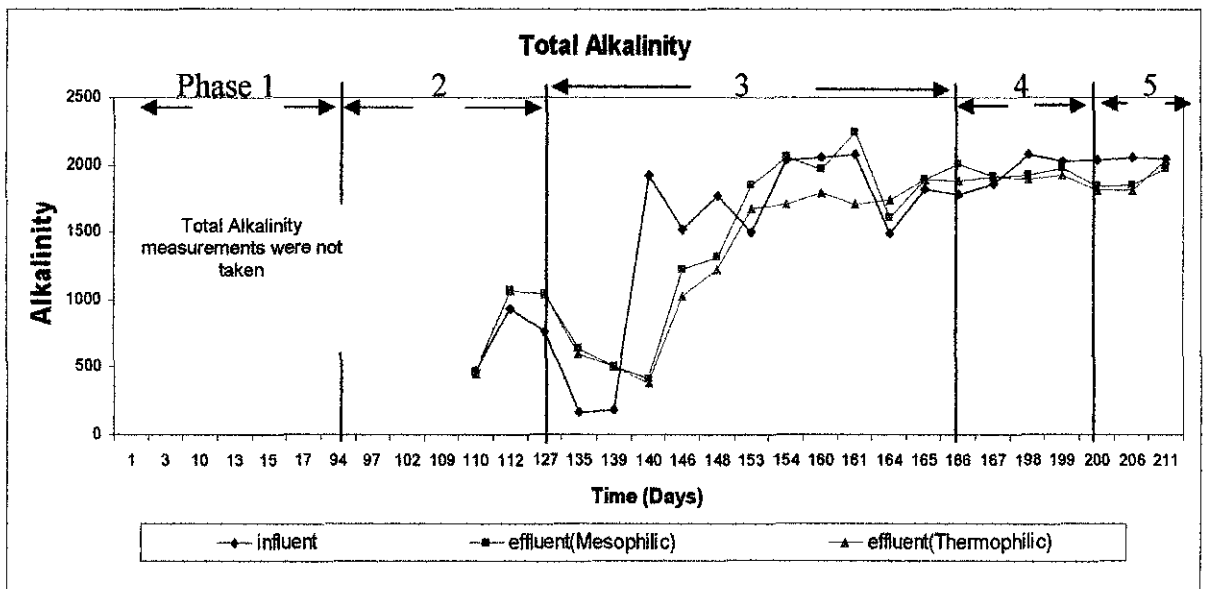
Figure 4.7: Alkalinity graph versus time (days)

Most anaerobic bacteria perform well within pH range of 6.8 to 7.2. Digester stability is enhanced by a high alkalinity concentration. The pH of the influent was controlled in the range of 7 to 8. Overall results showed that the pH of thermophilic effluent is higher compared to mesophilic effluent (Figure 4.8).
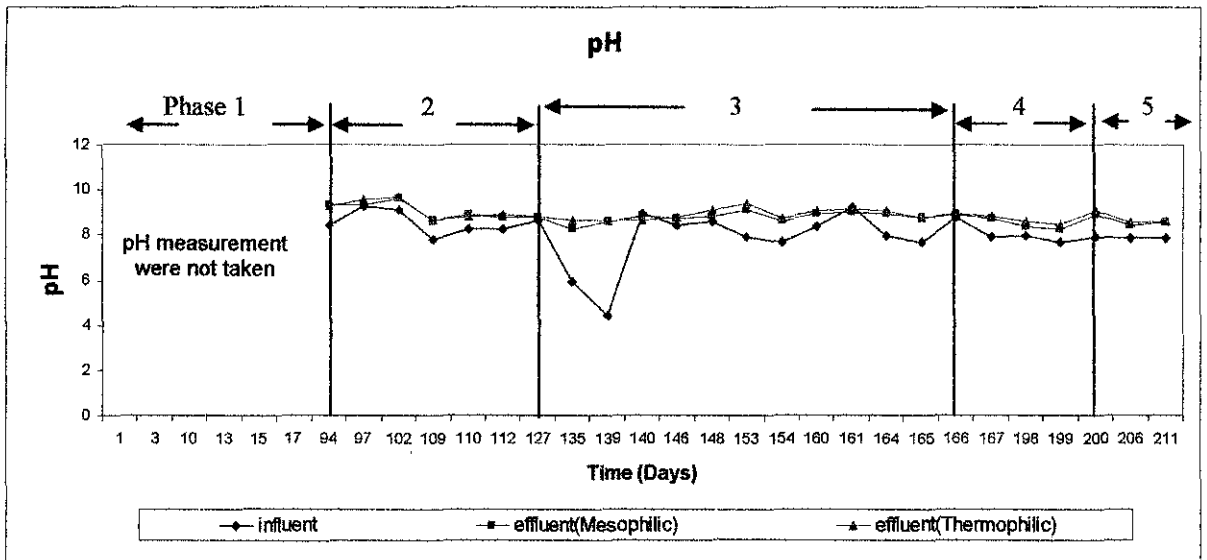
Figure 4.8: pH graph versus time (days)

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

As conclusion, at low and high strength concentration of wastewater, the mesophilic reactor has higher COD removal efficiency compared to thermophilic reactor. The methane produced by the mesophilic reactor is slightly higher than the thermophilic reactor. Organic loading rate and hydraulic retention time affect the percentage of COD removal.

From the lab results, it can be concluded that anaerobic treatment can treat pharmaceutical wastewater with high COD removal. By proper control, methane produced can be used as alternative energy source to generate the factory. Anaerobic treatment using UASB is not common in Malaysia but have already been applied in other developed as well as developing countries. It can produce methane which can be used as an alternative energy source.

5.2 Recommendations

For better performance of removal, the introduction of 2 stage treatment can be implemented with the combination of anaerobic and aerobic treatment. Thus the nutrient discharged from anaerobic reactor can be used as nutrient for the aerobic process.

For future recommendation, the monitoring of the reactor performance should be done in details to avoid error or accident that may cause reactor to failed or unable to

operate properly. The Volatile Fatty Acids test should be done so the process of anaerobic digestion to get better understanding of the reactor performance.

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(http://www.uem.com.my/biz_health.php)

(http://www.uemgroup.com)

APPENDIX A

PARAMETER LIMITS OF EFFLUENT OF STANDARDS A AND B

Parameter	Unit	Standard	
		A	B
(1)	(2)	(3)	(4)
(i) Temperature	°C	40	40
(ii) pH Value	-	6.0-9.0	5.5-9.0
(iii) BOD_5 at 20°C	mg/l	20	50
(iv) COD	mg/l	50	100
(v) Suspended Solids	mg/l	50	100
(vi) Mercury	mg/l	0.005	0.05
(vii) Cadmium	mg/l	0.01	0.02
(viii) Chromium, Hexavalent	mg/l	0.05	0.05
(ix) Arsenic	mg/l	0.05	0.10
(x) Cyanide	mg/l	0.05	0.10
(xi) Lead	mg/l	0.10	0.5
(xii) Chromium, Trivalent	mg/l	0.20	1.0
(xiii) Copper	mg/l	0.20	1.0
(xiv) Manganese	mg/l	0.20	1.0
(xv) Nickel	mg/l	0.20	1.0
(xvi) Tin	mg/l	0.20	1.0
(xvii) Zinc	mg/l	2.0	2.0 [Am P.U.(A) 398/2000]
(xviii) Boron	mg/l	1.0	4.0
(xix) Iron (Fe)	mg/l	1.0	5.0
(xx) Phenol	mg/l	0.001	1.0
(xxi) Free Chlorine	mg/l	1.0	2.0
(xxii) Sulphide	mg/l	0.50	0.50
(xxiii) Oil and Grease	mg/l	Not detectable	10.0

FIFTH SCHEDULE

ENVIRONMENTAL QUALITY ACT 1974

ENVIRONMENTAL QUALITY (SEWAGE AND INDUSTRIAL EFFLUENTS) REGULATIONS 1978

[Regulation 8 (4)]
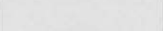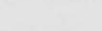
LIST OF PARAMETERS THE LIMITS OF WHICH TO BE SPECIFIED.

(i) Ammoniacal Nitrogen

(ii) Sulphate

(iii) Chloride

(iv) Cobalt

(v) Colour

(vi) Detergents, Anionic

(vii) Fluoride (as F)

(viii) Molybdenum

(ix) Nitrate Nitrogen

(x) Phosphate (as P)

(xi) Polychlorinated Biphenyls

(xii) Selenium

(xiii) Silver

(xiv) Beryllium

(xv) Vanadium

(xvi) Radioactive Material

(xvii) Pesticides, fungicides, herbicides, insecticides, rodenticides, fumigants or any other biocides or any other chlorinated hydrocarbons

(xviii) A substance that either by itself or in combination or by reaction with other waste or refuse may give to any gas, fume or odour or substance which causes or likely to cause pollution.

Project Milestone

ID	Task Name	Start	Finish	Duration	Jan 2008					Feb 2008				Mar 2008				Apr 2008	
					12/30	1/6	1/13	1/20	1/27	2/3	2/10	2/17	2/24	3/2	3/9	3/16	3/23	3/30	4/6
1	Submission of first draft	12/24/2007	4/1/2008	14.4w															
2	Submission of progress report 1	12/24/2007	2/8/2008	7w															
3	Lab work	12/24/2007	4/11/2008	16w															
4	Cod	12/24/2007	4/8/2008	15.4w															
5	Alkalinity	12/31/2007	4/11/2008	15w															
6	pH	12/24/2007	4/11/2008	16w															
7	Ammonia	2/1/2008	4/11/2008	10.2w															
8	Phosphorus	12/24/2007	4/10/2008	15.8w															
9	VSS/TSS	1/1/2008	4/9/2008	14.4w															
10	Gas production	1/1/2008	4/8/2008	14.2w															

Legend:

■		
Scheduled	Actual	Current

APPENDIX C

51

JOB SAFETY ANALYSIS (JSA)

Title of Job Operation	Chemical Oxygen Demand	Date	7/1/2008	Ref No	FYP2/JSA/001
Title of Person Who Does The Job	Muhamad Farhan	**Employee Observed**	none		
Location	Building 14				
Department/Program	Civil Engineering	**Prepared By**	Muhamad Farhan Abd Rahim		
Section/ Lab	Environmental Lab	**Approved by**	Ap Dr Hasnain		

Sequence of Basic Job Step	Potential Accident or Hazard	Recommended Safe Job Procedure
1. Sampling	1.1 Spill material to body	1.1.1 Wear proper ppe (lab coat)
2. Shaking the vials	2.1 Dropping the vials	2.1.1 Properly hold the vials when shake
3. Heating for 2 hours	3.1 Skin burning (finger)	3.1.1 Wear proper glove
		3.1.2 Close the cover after insert the vial in the heat
		3.1.3 Wait for the vial to cool down after two hour in proper vial holder

Title of Job Operation	Total Phosphate	Date	11/1/2008	Ref No	FYP2/JSA/002
Title of Person Who Does The Job	Muhamad Farhan	Employee Observed	none		
Location	Building 14				
Department/Program	Civil Engineering	Prepared By		Muhamad Farhan Abd Rahim	
Section/ Lab	Environmental Lab	Approved by		Ap Dr Hasnain	

Sequence of Basic Job Step	Potential Accident or Hazard	Recommended Safe Job Procedure
1. Sampling	1.1 Spill material to body	1.1.1 Wear proper ppe (lab coat)
2. Pouring potassium persulfate powder into vial	2.1 Inhaling the powder	2.1.1 Wear mask if necessary
		2.1.2 Keep distance between the vials and properly pour the powder inside the vials.
3. Heating for ½ hours	3.1 Skin burning (finger)	3.1.1 Wear proper glove
		3.1.2 Close the cover after insert the vial in the heat
		3.1.3 Wait for the vial to cool down after ½ hour in proper vial holder
4. Pouring NaOH inside the vial	1.4 Spill material to body	1.4.1 Wear proper ppe (lab coat)
5. Pouring Phos Ver 3 powder inside vial	1.5 Inhaling the powder	1.5.1 Wear mask if necessary
		1.5.2 Keep distance between the vials and properly pour the powder inside the vials.
6. Shaking	1.6 Dropping the vial	1.6.1 Hold properly the vial when shake

JOB SAFETY ANALYSIS (JSA)

Title of Job Operation	Ammonia Nitrogen test (Nessler Method)	Date	11/2/2008	Ref No	FYP2/JSA/003
Title of Person Who Does The Job	Muhamad Farhan	Employee Observed	none		
Location	Building 14				
Department/Program	Civil Engineering	Prepared By		Muhamad Farhan Abd Rahim	
Section/ Lab	Environmental Lab	Approved by		Ap Dr Hasnain	

Sequence of Basic Job Step	Potential Accident or Hazard	Recommended Safe Job Procedure
1. Sampling	1.1 Spill material to body	1.1.1 Wear proper ppe (lab coat)
2. Add 3 drops polyvinyl alcohol	2.1 Spill material to body	2.1.1 Wear proper ppe (lab coat)
		2.1.2 Keep distance between the conical flask and properly drop the polyvinyl alcohol inside the conical flask
3. Insert 2ml of nessler reagent	3.1 Spill material to body	3.1.1 Wear proper ppe (lab coat)
		3.1.2 Keep distance between the conical flask and properly drop the polyvinyl alcohol inside the conical flask
4. Shaking	4.1 Dropping the conical flask causing the glass to scatter	4.1.1 Properly and slowly mixing the sample

JOB SAFETY ANALYSIS (JSA)

Title of Job Operation	Alkalinity	Date	16/1/2008	Ref No	FYP2/JSA/004
Title of Person Who Does The Job	Muhamad Farhan	**Employee Observed**		none	
Location	Building 14				
Department/Program	Civil Engineering	**Prepared By**		Muhamad Farhan Abd Rahim	
Section/ Lab	Environmental Lab	**Approved by**		Ap Dr Hasnain	

Sequence of Basic Job Step	Potential Accident or Hazard	Recommended Safe Job Procedure
1. Sampling	1.1 Spill material to body	1.1.1 Wear proper ppe (lab coat)
2. Filling the sulphuric acids inside the burette	2.1 Spill material to body	2.1.1 Wear proper ppe (lab coat)
	2.2 Spill material to eye	2.2.1 Wear goggle if necessary
	2.3 Spill material to body	2.3.1 Keep distance between the burette and use appropriate equipment
3. Shaking	3.1 Dropping the conical flask causing the glass to scatter	3.1.1 Properly and slowly mixing the sample

Title of Job Operation	Total Suspended Solids and Volatile Solids	Date	16/2/2008	Ref No	FYP2/JSA/005
Title of Person Who Does The Job	Muhamad Farhan	Employee Observed	none		
Location	Building 14				
Department/Program	Civil Engineering	Prepared By		Muhamad Farhan Abd Rahim	
Section/ Lab	Environmental Lab	Approved by		Ap Dr Hasnain	

Sequence of Basic Job Step	Potential Accident or Hazard	Recommended Safe Job Procedure
1. Sampling	1.1 Spill material to body	1.1.1 Wear proper ppe (lab coat)
2 Heating the sample for 1 hours inside the oven	2.1 Skin burning	2.1.1 Wear proper glove
3 Burning the sample inside the furnace at 500 degree Celsius	3.1 Skin burning	3.1.1 Wear proper glove
		3.1.2 Wait for the sample to cool down before taking it out

Date	Day	COD			Phosphorus			Ammonia Nitrogen NH3-N			pH		
		I	M	T	I	M	T	I	M	T	I	M	T
21/9/2007	1	532											
23/9/2007	3	472	81	633									
30/9/2007	10	469	81	633									
3/10/2007	13	361	79	90									
5/10/2007	15	356	117	161									
7/10/2007	17	483	71	160									
7/1/2008	94	316	55	92							8.43	9.31	9.27
9/1/2008	97	297	69	115							9.29	9.32	9.57
11/1/2008	102	450	49	90	6.17	15.9	17				9.08	9.62	9.65
15/1/2008	109	373	56	79	3.97	12.13	14.27				7.78	8.64	8.67
16/1/2008	110	402	46	57	1.85	9.25	11.27				8.244	8.877	8.789
18/1/2008	112	352	70	87	3.9	13.15	14.77				8.22	8.78	8.85
4/2/2008	127	473	64	84	9.62	40.5	38.3				8.633	8.77	8.789
11/2/2008	135	1850	142	133	11.73	24.43	29.47	2.35	6.03	3.6	5.93	8.24	8.65
14/2/2008	139	1743	89	153	10.17	18.5	21.77	9.2	12	12	4.46	8.59	8.62
15/2/2008	140	1837	165	283	30	33.33	44.33	3.53	7.83	4.1	8.925	8.817	8.658
21/2/2008	146	2127	187	211	26.33	34	38.5	2.77	7.37	4.2	8.422	8.722	8.743
23/2/2008	148	1695	155	171	12.43	14.27	16.53	4.3	6.23	3.7	8.56	8.79	9.11
28/2/2008	153	2157	249	230	11.3	14.87	18.43	4.07	10.3	8.8	7.92	9.09	9.37
29/2/2008	154	1695	205	299	10.37	13.77	17.27	5.4	10.63	7.5	7.678	8.654	8.734
6/3/2008	160	1677	272	393	8.83	16.13	20.93	2.13	10.87	6.33	8.34	8.91	9.07
7/3/2008	161	1617	262	334	14.77	16.1	18.93	9.33	10.37	7.03	9.22	9	9.13
10/3/2008	164	1860	249	386	13.8	17.37	19.57	7.7	10.07	7.43	7.95	8.95	9.03
11/3/2008	165	2010	268	397	12.3	17.93	20.63	4.43	11.57	9.83	7.67	8.73	8.78
12/3/2008	166	1903	270	384	9.7	16.4	18.8	8.77	11.8	9.83	8.77	8.85	8.95
21/3/2008	167	1995	1260	306	13.5	33.2	19.4	5.2	32.2	9.6	7.9	8.69	8.81
15/4/2008	198	1983	812	314	12.63	48.3	19.03	2.57	32.37	11.93	7.94	8.36	8.59
16/4/2008	199	1820	889	335	12.27	82	19.57	5.73	36.67	13.6	7.698	8.259	8.494
17/4/2008	200	1780	749	303	15.27	78	18.13	13.37	17.67	7.67	7.896	8.858	9.036
23/4/2008	206	1690	595	356	12.97	32.53	20.13	18.4	14.43	13.03	7.826	8.431	8.509
28/4/2008	211	1600	490	325	14.2	30.75	22.27	7.8	5.93	6.17	7.828	8.521	8.604

Phenol Alkalinity			Total Alkalinity			Gas (methane)		COD removal		%removal		HRT (days)	OLR
I	M	T	I	M	T	M	T	M	T	M	T		
												5	0.11
								391		82.8		5	0.09
								388		82.7		5	0.09
								282	271	78.1	75.1	5	0.07
								239	195	67.1	54.8	5	0.07
								412	323	85.3	66.9	5	0.10
						320	224	261	224	82.7	71	3	0.11
						208	80	228	182	76.8	61.2	3	0.1
						112	48	400	359	89	79.9	3	0.15
						64	48	317	294	85	78.7	3	0.12
16	76	48	475	467	457	112	32	356	346	88.6	85.9	3	0.13
0	72	107	940	1075	1073	112	60	282	265	80.2	75.4	3	0.12
84	84	88	760	1044	1040	352	240	409	389	86.5	82.2	3	0.16
0	16	15	160	640	592	160	144	1708	1717	92.3	92.8	5	0.37
0	48	40	181	507	507	288	112	1654	1590	94.9	91.2	5	0.35
88	48	40	1920	416	384	224	136	1671	1554	91	84.6	5	0.37
56	56	92	1520	1224	1028	224	163	1939	1916	91.2	90.1	5	0.43
67	59	88	1771	1317	1224	224	208	1540	1524	90.8	89.9	5	0.34
0	92	120	1500	1848	1676	160	96	1908	1927	88.5	89.4	5	0.43
0	85	85	2038	2055	1711	180	150	1490	1396	87.9	82.4	5	0.34
86	85	72	2061	1960	1787	152	130	1404	1284	83.8	76.6	5	0.34
80	76	83	2075	2245	1704	112	144	1354	1283	83.8	79.3	5	0.32
0	104	72	1488	1608	1736	180	128	1611	1474	86.6	79.3	5	0.37
0	72	52	1820	1896	1888	128	80	1742	1613	86.7	80.2	5	0.4
48	72	48	1784	2008	1880	144	128	1634	1519	85.8	79.8	5	0.38
			1860	1912	1912	256	128	735	1689	36.8	84.7	4	0.5
			2080	1928	1890	52	130	1171	1669	59.1	84.2	4	0.5
			2022	1980	1920	48	128	931	1485	51.2	81.6	4	0.46
			2040	1840	1808	64	112	1031	1477	57.9	83	4	0.45
			2056	1856	1812	160	176	1095	1334	64.8	78.9	3	0.56
			2048	1972	2040	80	32	1110	1275	69.4	79.7	3	0.53