Determination of wastewater characteristics from Chemical Engineering Programme Laboratories

by

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CERTIFICATION OF APPROVAL

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Approved by,

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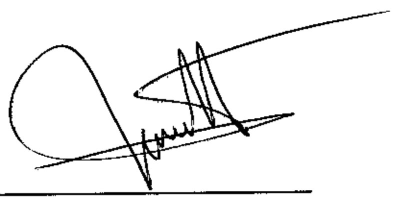
CERTIFICATION OF ORIGINALITY

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.



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ABSTRACT

University Technology PETRONAS, UTP, has several wastewater treatment plants treating wastewater generated within the university campus. A small wastewater treatment plant located behind Chemical Engineering Programme Buildings is the subject matter for this project. Recently, issue on whether the existing Effluent Treatment Plant for Chemical Engineering Programme Buildings is suitable with the characteristics of the waste produced has been raised.

Several characteristics of the wastewater influent to the wastewater plant were analyzed and as a result all the tested parameters except for chromium (VI) content showed that the existing plant is suitable with the characteristics of the waste produced. Hexavalent chromium concentration was found to be in the range of 0.05 – 0.086 mg/L, which is more than the allowable limit of 0.05 mg/L.

ACKNOWLEDGEMENT

Firstly, I would like to express my deep appreciation to Dr. Hilmi Mukhtar, Head of Chemical Engineering, for his help and support in completing this project and eventually successfully graduating from UTP.

Secondly, I would like to express my appreciation to the most important person in this project, my supervisor Mr. Azizul Buang. His guidance, encouragement, advice, useful comments, and feed back have made it possible to complete this project.

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CHAPTER 1
INTRODUCTION

1.1 Background of Study

Every community produces both liquid and solid wastes and air emissions. The liquid waste – wastewater – is essentially the water supply of the community after it has been in a variety of applications. From the viewpoint of sources of generation, wastewater may be defined as a combination of the liquid or water-carried wastes removed from residences, institutions, and commercial and industrial establishments, together with such groundwater, surface water, and storm water as may be present.

When untreated wastewater accumulates and is left as it is, the decomposition of the organic matter it contains will lead to nuisance conditions including the production of malodorous gases. In addition, untreated wastewater contains numerous microorganisms that cause disease to human body. Wastewater also contains nutrients, which can stimulate the growth of aquatic plants, and may contain toxic compounds or compounds that potentially may be mutagenic or carcinogenic. For these reasons, the immediate and nuisance-free removal of wastewater from its sources of generation, followed by treatment, reuse, or dispersal into the environment is necessary to protect public health and the environment.

As being related to the subject of this project, concern has been raised about effluents from university due to the complexity of chemical mixtures that are used and discharged from laboratory facilities.

1.2 Problem statement

University Technology PETRONAS, UTP, has several wastewater treatment plants treating wastewater generated within the university campus. A small wastewater treatment plant located behind Chemical Engineering Programme Buildings is the subject matter for this project. Recently, issue on whether the existing Effluent Treatment Plant for Chemical Engineering Programme Buildings is suitable with the characteristics of the waste produced has been raised. It is reported that the plant has not been designed to treat scheduled waste such as cyanide and chromium.

The concern is that based on the types of experiments conducted in the laboratories of those buildings, such scheduled waste may be present in the effluent.

CHAPTER 2
OBJECTIVE AND SCOPE OF STUDY

Objectives of this project are:

1. To determine physical characteristics of wastewater such as turbidity, total suspended solids (TSS).
2. To determine the chemical characteristics of wastewater such as pH.
3. To determine the organic contaminants in the effluent via COD analysis and presence of toxic chemicals, namely Cr^{6+}.
4. To check whether the existing plant is suitable for treating such waste.

The characteristics of the waste, which depends on the types of chemical used and the volume being consumed, are difficult to be analyzed completely. On that note, the scope of the project has been focused only on several important characteristics of wastewater. These characteristics are based on

1. Typical experiment conducted are COD analysis, hence Cr^{6+} determination is important.
2. Standard A/B requirement of EQA (Sewage and Industrial Effluents) Regulations 1979.

CHAPTER 3
LITERATURE REVIEW

3.1 Turbidity

Turbidity is a measure of the light-transmitting properties of water. It is a test used to indicate the quality of waste discharges and natural waters with respect to colloidal and residual suspended matter. The measurement of turbidity is based on comparison of the intensity of light scattered by a sample to the light scattered by a reference suspension under the same conditions (Standard Methods, 1998). Formazin suspensions are used as the primare reference standard the results of turbidity measurements are reported as nephelometric turbidity units (NTU). Colloidal matter will scatter or absorb light and thus prevent its transmission. It should be noted that the presence of air bubbles in the fluid will cause erroneous turbidity readings. In general, ther is no relationship between turbidity and the concentration of total suspended solids in untreated wastewater.

On of the problems with the measurement of turbidity is the high degree of variability observed, depending on the light source (incandescent light versus light-emitting diodes) and the method of measurements (reflected versus transmitted light). Another problem often encountered is the light-absorbing properties of the suspended material. For example, the turbidity of a solution of lampblack will essentially be equal to zero. As a result, it is almost impossible to compare turbidity values reported in the literature. However, turbidity readings at a given facility can be used for process control. Some on-line turbidity meters used to monitor the performance of microfiltration units are affected by the air used to clean the membranes.

<u>Factors Affecting Turbidity</u>

Because one of the primary factors affecting turbidity is toal suspended solids, the factors affecting TSS will also affect turbidity. In addition, organic matter contributes to turbidity.

High Flow Rates

The flow rate of a water body is a primary factor influencing turbidity concentrations. Fast running water can carry more particles and larger-sized sediment. Heavy rains can pick up sand, silt, clay, and organic particles from the land and carry it to surface water. A change in flow rate also can affect turbidity; if the speed or direction of the water current increases, particulate matter from bottom sediments may be resuspended.

Soil Erosion

Soil erosion is caused by disturbance of a land surface. Soil erosion can be caused by Building and Road Construction, Forest Fires, Logging, and Mining. The eroded soil particles can be carried by stormwater to surface water. This will increase the turbidity of the water body.

Urban Runoff

During storm events, soil particles and debris from streets and industrial, commerical, and residential areas can be washed into streams. Because of the large amount of pavement in urban areas, natural settling areas have been removed, and sediment is carried through storm drains to creeks and rivers.

Wastewater and Septic System Effluent

The effluent from Wastewater Treatment Plants (WWTPs) can add suspended solids and organic material to a stream. The wastewater from our houses contains food residue, human waste, and other solid material that we put down our drains. Most of the solids and organic material are removed from the water at the WWTP before being discharged to the stream, but treatment can't eliminate everything.

Decaying Plants and Animals

As plants and animals present in a water body die and decay, suspended organic particles are released and can contribute to turbidity.

Bottom-Feeding Fish

Bottom-feeding fish (such as carp) can stir up sediments as they remove vegetation. These sediments can contribute to turbidity.

Algal Blooms

Algal blooms can contribute to turbidity. Algal production is enhanced when nutrients are released from bottom sediments during seasonal turnovers and changes in water current.

Flooding

As flood waters recede, they will bring along inorganic and organic particles from the land surface, and contribute this to the stream.

3.2 Total Suspended Solids (TSS)

Wastewater contains a variety of solid materials varying from rags to colloidal material. In the characterization of wastewater, coarse materials are usually removed before the sample is analyzed for solids. TSS is a common parameter used in defining a municipal or industrial wastewater. Operational efficiency of various treatment units is defined by solids removal in settling tank and volatile solids reduction in sludge digestion. Total solids, total residue on evaporation is the term applied to material left in a dish after evaporation of a sample of water or wastewater and subsequent drying in an oven. The term suspended solids refer to the matter that is retained and passed through a standard glass-fiber filter. High concentrations of suspended solids can cause many problems for stream health and aquatic life.

High TSS can block light from reaching submerged vegetation. As the amount of light passing through the water is reduced, photosynthesis slows down. Reduced rates of photosynthesis causes less dissolved oxygen to be released into the water by plants. If light is completely blocked from bottom dwelling plants, the plants will stop producing oxygen and will die. As the plants are decomposed, bacteria will use up even more oxygen from the water. Low dissolved oxygen can lead to fish kills. High TSS can also cause an increase in surface water temperature, because the suspended particles absorb heat from sunlight. This can cause dissolved oxygen levels to fall even further (because warmer waters can hold less DO), and can harm aquatic life in many other ways, as discussed in the temperature section. (Mitchell and Stapp, 1992; KanCRN website)

The decrease in water clarity caused by TSS can affect the ability of fish to see and catch food. Suspended sediment can also clog fish gills, reduce growth rates, decrease resistance to disease, and prevent egg and larval development. When suspended solids settle to the bottom of a water body, they can smother the eggs of fish and aquatic insects, as well as suffocate newly hatched insect larvae. Settling sediments can fill in spaces between rocks which could have been used by aquatic organisms for homes. (Mitchell and Stapp, 1992; GREEN Hands-On Center website)

High TSS in a water body can often mean higher concentrations of bacteria, nutrients, pesticides, and metals in the water. These pollutants may attach to sediment particles on the land and be carried into water bodies with storm water. In the water, the pollutants may be released from the sediment or travel farther downstream (Federal Interagency Stream Restoration Working Group, 1998).

High TSS can cause problems for industrial use, because the solids may clog or scour pipes and machinery.

To measure TSS, the water sample is filtered through a pre-weighed filter. The residue retained on the filter is dried in an oven at 103 to 105° C until the weight of the filter no longer changes. The increase in weight of the filter represents the total suspended solids. TSS can also be measured by analyzing for total solids and subtracting total dissolved solids.

Factors Affecting Total Suspended Solids

High Flow Rates

The flow rate of the water body is a primary factor in TSS concentrations. Fast running water can carry more particles and larger-sized sediment. Heavy rains can pick up sand, silt, clay, and organic particles (such as leaves, soil, tire particles) from the land and carry it to surface water. A change in flow rate can also affect TSS; if the speed or direction of the water current increases, particulate matter from bottom sediments may be resuspended.

Soil Erosion

Soil erosion is caused by disturbance of a land surface. Soil erosion can be caused by Building and Road Construction, Forest Fires, Logging, and Mining. The eroded soil particles can be carried by stormwater to surface water. This will increase the TSS of the water body.

Urban Runoff

During storm events, soil particles and debris from streets and industrial, commercial, and residential areas can be washed into streams. Because of the large amount of pavement in urban areas, infiltration is decreased, velocity increases, and natural settling areas have been removed. Sediment is carried through storm drains directly to creeks and rivers.

Wastewater and Septic System Effluent

The effluent from Wastewater Treatment Plants (WWTPs) can add suspended solids to a stream. The wastewater from our houses contains food residue, human waste, and other solid material that we put down our drains. Most of the solids are removed from the water at the WWTP before being discharged to the stream, but treatment can't eliminate everything.

Decaying Plants and Animals

As plants and animals decay, suspended organic particles are released and can contribute to the TSS concentration.

Bottom-Feeding Fish

Bottom-feeding fish (such as carp) can stir up sediments as they remove vegetation. These sediments can contribute to TSS.

3.3 pH

The hydrogen-ion concentration is an important quality parameter of wastewaters. The usual means of expressing the hydrogen-ion concentration is as pH, which is defined as the negative logarithm of the hydrogen-ion concentration.

The concentration range suitable for the existence of most biological life is quite narrow and critical (typically 6 to 9). Wastewater with an extreme concentration of hydrogen ion is difficult to treat by biological means, and if the concentration is not altered before discharge, the wastewater effluent may alter the concentration in the natural waters. For treated effluents discharged to the environment the allowable pH range usually varies from 6.5to 8.5.

The pH of a solution is measured either by an indicator dye or by a pH meter and an electrode system whose voltage output is proportional to the active acid ($H3O+$) concentration in solution.

Certain organic dye solutions change color over a relatively small pH range. These are called indicator solutions. They can be used to indicate the approximate pH of a solution. By adding a few drops of a phenolphthalein indicator to a solution one can tell if the pH of the solution has a pH greater than 9 by the red color present, or a pH less than 9 by the lack of color. Other dye materials can be chosen whose color changes indicate other pH ranges. For example, phenol red changes at pH 8, bromthymol blue at pH 7, and bromphenol blue at pH 4.

For convenience, these dyes are often deposited on a strip of paper. When a drop of solution to be tested is placed on the paper, the resulting color change is indicative of the approximate pH of the test solution. Dye indicator solutions or paper have the advantage of being quite inexpensive, very portable, and often suitable where only an approximate pH measurement is needed. On the other hand, where precise measurements are needed and / or the solution to be measured is colored, a pH meter is required. Accordingly, pH meter and electrode systems have been developed which respond in a precise manner to the pH of a solution.

To measure pH one can use any number of readily available ($25 – $100) pH probes. A pH probe acts like a battery that proportionately generates positive DC voltage for low pH, nothing for pH 7, and negative voltages for high pH values. So, all we have to do is measure this voltage and convert it to pH units.

But there are two problems involved. One problem is that pH is temperature sensitive, with the output voltage ranging from 54 millivolts per pH unit at zero degrees centigrade up to 74 millivolts per pH unit at 100° C. This means that we have to manually vary the gain or conversion constant of our pH measurement to be able to correct for temperature of the solution being measured.

The second problem is a bit more complex and explains the previously high cost of pH instruments. The source impedance of our pH probe is 15 megohms for the "low–impedance" probes and ranges upwards into hundreds of megohms for special units. In order to measure pH, our voltage amplifier must have an input impedance that is very high compared with 15 megohms. Here is where CMOS electronics has come to the rescue, producing accurate and inexpensive pH meters.

3.4 Chemical Oxygen Demand (COD)

COD is used as a measurement of pollutants in natural and waste waters to assess the strength of discharged waste such as sewage and industrial effluent waters. It is normally measured in both municipal and industrial wastewater treatment plants and gives an indication of the efficiency of the treatment process. COD is measured in both influent and effluent water (before and after treatment). The efficiency of the treatment process is normally expressed as COD Removal, measured as a percentage of the organic matter removed during the cycle.

Untreated wastewater is generally rich in organic matter. This organic matter feeds the bacteria and algae normally present in healthy water sources. The presence of excessive amounts of nutrients discharged as a result of untreated wastewater will cause an increase in concentration of both bacteria and algae within the surface water. Beside organic matter, wastewater also contains both organic and oxidizable inorganic compounds. These organic and inorganic compounds directly and indirectly consume the available oxygen present in the ecosystem. This process is called eutrification and will eventually kill off other living organisms (plants, animals, & insects) in the aquatic system.

Governments strictly control these oxygen demanding pollutants by setting standards for maximum levels of "oxygen demand" for all discharged wastewaters. There are different methods known for measuring the oxygen demand but BOD (Biological Oxygen Demand) and COD (Chemical Oxygen Demand) are the most widely accepted methods. The COD test has many advantages over the BOD method. The COD test is more accurate and faster than the BOD method. The BOD method requires a five to seven day incubation period and is more subject to operator errors. The COD method usually takes under three hours and is easy to perform by technical and non-technical personnel alike.

3.5 Chromium Hexavalent

Chromium occurs mainly in three forms. Metallic chromium (Cr[0]) is a steel-gray solid with a high melting point that's used to make steel and other alloys. Chromium metal does not occur naturally; it is produced from chrome ore. Trivalent chromium (Cr[III]) occurs naturally in rocks, soil, plants, animals, and volcanic emissions. This form is believed by many to play a nutritional or pharmaceutical role in the body, but its mechanism of action is unknown. Cr(III) is used industrially as a brick lining for high-temperature industrial furnaces and to make metals, metal alloys, and chemical compounds. Hexavalent chromium (Cr[VI]) is produced industrially when Cr(III) is heated in the presence of mineral bases and atmospheric oxygen (for instance, during metal finishing processes). It is this third form of chromium that has proven to be of the greatest occupational and environmental health concern.

Cr(VI) compounds are emitted into the air, water, and soil by a number of different industries. In the air, chromium compounds are present mainly as fine dust particles that eventually settle over the land and water.

The *Report on Carcinogens,* published by the National Toxicology Program, indicates that typical tap water can contain 0.4-8.0 μg per liter (L) total chromium, and that chromium in rivers and lakes usually falls between 1 and 10 μg/L. Cr(VI) by itself is not regulated in drinking water. The U.S. Environmental Protection Agency (EPA) regulates only total chromium in drinking water and has set a maximum contaminant level of 100 μg/L (more stringent state limits are often set at half that amount).

Research on Cr(VI) is ongoing, but there are still many questions to be answered about the human health effects of this industrial heavyweight. However, with a growing base of results from recent studies, the ever-expanding field of molecular epidemiology, and growing cooperation between industry, research, and regulatory agencies, maybe it won't take another 100 years to find the answers.

3.6 Types of chemicals present in the effluent

In Chemical Engineering Programme, the syllabus combines both theoretical and laboratory works to enhance the knowledge of the students. Based on the types of experiments and the frequency of the laboratory session, the accumulation of chemicals over a period of time could result in a major concern and should not be overlooked.

Below is tabulated the types of chemicals in effluent discharged from Chemical Engineering Programme Buildings and their estimated volume annually.

Table 3.1 Types of chemicals in effluent stream discharged from Chemical Engineering Programme Buildings and their estimated volume annually

Digestion Solution for COD	5 Liter
Acetone	240 mL
Methanol	240 mL
Sodium Hydroxide 0.02 N	500 mL
Sulfuric Acid	500 mL
Hydrochloric Acid 1N	100 mL
Calcium Hydroxide	500 mL
Chromium III Chloride	250 mL
Copper I Chloride	250 mL
Copper II Nitrate	250 mL
Zinc Nitrate	250 mL
Magnesium Hydroxide	250 mL
Digestion for COD, Low Range Potassium Di Chromate	500 mL
Digestion for COD, High Range Potassium Di Chromate	500 mL
Selenium Catalyst	50 mL

3.7 Description of the existing treatment plant

During the set-up and establishment of the new campus of UTP, the designer and builder have constructed a dedicated waste treatment plant for the Chemical Engineering Programme. Based on the generic treatment stages for wastewater, the contractor has built a treatment plant which consists of these four treatment stages:

1. Pre-treatment: At the first stage, oil and grease is removed from raw effluent.
2. Primary Treatment: Once the oil and grease is removed, the waste is treated by chemical coagulation and flocculation to remove suspended solids in the waste. pH is also adjusted at this stage. Any sludge created shall be removed by gravity sedimentation.
3. Secondary Treatment: After the chemical-physical process, collected sludge is treated with activated carbon by adsorption process.

In figure below is shown the schematic – block flow diagram of the wastewater treatment system.

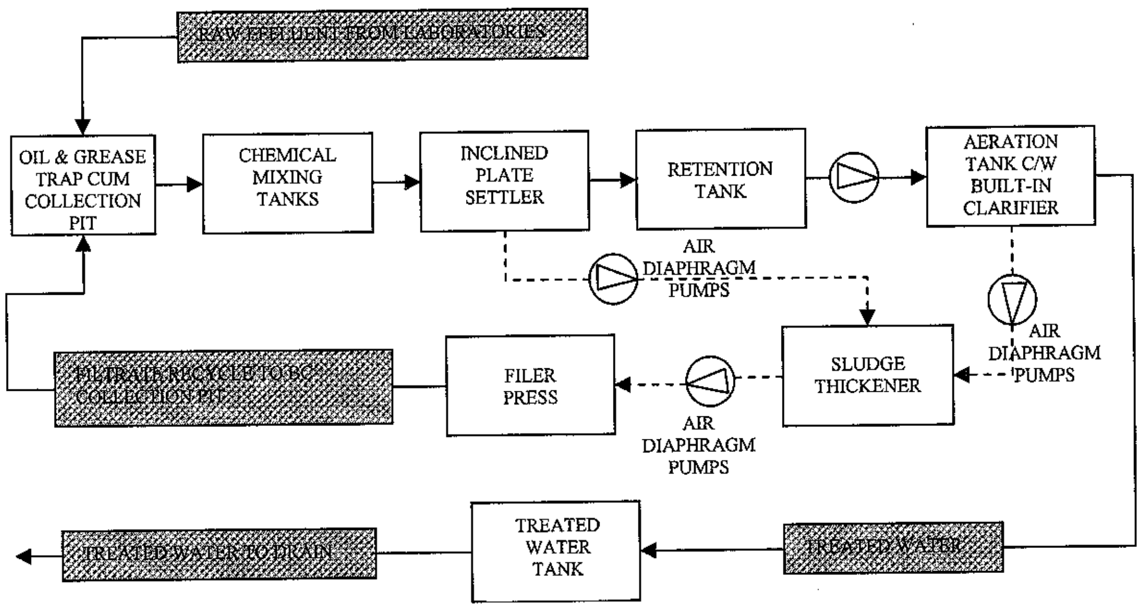

Figure 3.1 Schematic-block flow diagram of the existing wastewater treatment plant

Referring to the above plant design/layout, there is no suitable treatment method incorporated for converting toxic Cr^{6+} to Cr^{3+}.

Table below presents standards for wastewater under the EQA (Sewage and Industrial Effluent) Regulation 1979.

Table 3.2 Standards for wastewater under the EQA (Sewage and Industrial Effluent) Regulation 1979.

Parameter	**Unit**	**Standard A**	**Standard B**
Temperature	°C	40	40
pH	-	6 – 9	5.5 – 9
BOD_5	mg/L	20	50
COD	mg/L	50	100
TSS	mg/L	50	100
Mercury	mg/L	0.005	0.05
Cadmium	mg/L	0.01	0.02
Chromium (VI)	mg/L	0.05	0.05
Cyanide	mg/L	0.05	0.1
Plumbum	mg/L	0.1	0.5
Chromium (III)	mg/L	0.5	1.0
Silver	mg/L	0.2	1.0
Manganese	mg/L	0.2	1.0
Nickel	mg/L	0.2	1.0
Zinc	mg/L	1.0	1.0
Iron	mg/L	1.0	5.0
Oil and Grease	mg/L	10.0	10.0

CHAPTER 4
METHODOLOGY

Number of samples from the middle and the bottom of the wastewater tank were used to measure abovementioned properties of the wastewater. Below are methodology adopted for the experiments.

4.1 Turbidity

The turbidity test was conducted using the DR/4000 1-inch cell adapter.

10 ml wastewater samples were used each being filled in a sample cell. Both samples then were put inside the cell holder and the light shield is closed. Turbidity values were obtained through the measurement of light deflection by the DR/4000 unit.

4.2 Total Suspended Solids

The determination of TSS was made by the use of analytical balance, Gravimetric analysis. Gravimetric analysis is where the measurements require some sort of crucibles or dishes to hold the residue.

TSS was measured by weighing the filter after filtration and drying at 103 to 105 degree C and then subtracting the weight of the clean filter from the result. Dividing this result by the volume of the sample would give the TSS concentration.

4.3 pH

A set of portable pH meter was used to determine the pH. The probe was immersed in the sample and the pH reading was recorded after the meter reading was stabilized.

4.4 COD

2ml of the samples was placed into a test tube containing potassium dichromate in sulfuric acid solution. The tube was then heated to enhance the oxidation process. The samples were tested for COD using the spectrophotometer.

4.5 Chromium Hexavalent

Hexavalent chromium is determined by the 1,5-Diphenylcarbonhydrazine method using a single dry powder formulation called Chroma Ver3 Chromium Reagent. This reagent contains an acidic buffer combined with 1,5-Diphenylcarbonhydrazine, which reacts to give a purple color when hexavalent chromium is present in a sample. Samples were put into a sample cell together with the reagent after 8 minutes the cell was put inside the DR/4000 cell holder. Results were obtained after the analysis by DR/4000 completed.

CHAPTER 5
RESULTS AND DISCUSSION

The table below shows the results of the analysis conducted. These figures are actually representing samples before treatment. That is they are of influent stream to the treatment plant.

Table 5.1 Several characteristics of wastewater influent to the treatment plant.

	Sample	TSS (mg/L)	Turbidity (NTU)	COD (mg/L)	pH	Chromimum Cr^{6+} (mg/L)
Bottom sample	1	8	4.36	31	7.7	0.050
	2	3	4.36	31	7.7	0.052
	3	8	4.36	31	7.7	0.086
Middle Sample	1	9	3.85	27	7.74	0.071
	2	7	3.85	27	7.74	0.072
	3	5	3.85	27	7.74	0.050

The TSS values of the effluent stream seem very low even if being compared with Standard A of the Regulation 1979 which is 50 mg/L.

The factors that contribute to the TSS value include usage of solid samples in experiments, washing and rinsing of glass wares.

The turbidity values of the samples are observed to be higher for the bottom sample. The value of 4.36 NTU may be due to the settling of large or heavy colloidal particles impurities.

The COD is also low compared to the Regulation 1979. Based on the experiments, usage of organic chemicals might be the main contributors to these values.

The analysis for Cr^{6+} would raise some concern since the value obtained is higher than the allowable limits. The results of the analysis showed level of Cr^{6+} between 0.05 and 0.086 mg/L.

Although these figures were basically from samples prior to the treatment plant but based on the design of the plant, such waste could not be treated.

CHAPTER 6
CONCLUSION & RECOMMENDATIONS

Based on the findings of the analysis and the design/layout of the treatment plant, no concern should be identified in terms of treatment for physical characteristics of the wastewater from Block 3, 4 and 5 such as TSS, COD and turbidity.

In terms of treating Cr^{6+}, which was determined to be present in a quite high level (compared to the Regulation 1979), there would be a major problem since such treatment method requires conversion of Cr^{6+} to Cr^{3+}. Such conversion can only be obtained through reduction and oxidation treatment method. In the existing plant, there is no such method available.

On this note, it is recommended that either

1. Cr^{6+} containing wastes in the laboratories should be separated at source, meaning that they should not be mixed with other types of wastewater generated.

or

2. the existing wastewater treatment plant should incorporate a stage where toxic Cr^{6+} should be converted to nontoxic Cr^{3+}. This could be done by adding glucose into the waste.

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