

## CHAPTER THREE : MATERIALS AND METHODS

### 3.1 Wastewater Samples

In this comparative study of performance of WWTPs in three rubber glove manufacturing factories, grab samples were collected from the respective factories. These factories were selected as they are manufacturing similar products. The WWTPs at these factories were observed to be well maintained and their performances have been satisfactory. In addition, the WWTPs for all the three factories of these factories have adopted similar design system for their WWTPs. The locations of these factories were indicated in Figure 3.1. Factory A is located in Kuala Pilah, Negeri Sembilan, while Factory B is located in Kuantan, Pahang and lastly Factory C in Sitiawan, Perak. Samples were collected at three sampling points along the WWTP process i.e. 1) from the equalisation basin, 2) at the outlet of DAF unit and 3) at the discharge sump after the secondary clarifier, as shown in Figure 3.2.

The samplings were carried out throughout the period of four months for Factory A and three months for Factory B and C. Samples were collected consecutively for 3 days in a week, with 3 samples at each sampling point. The samplings were done at an interval of half an hour. The samples were then mixed and collected in 1-liter sampling bottles, and were kept in icebox. It is not advisable to leave the samples at room temperature, as it will reduce the BOD, for instance an 8 hours period delay could reduce the BOD by as much as 40% (Jackson, 1993).

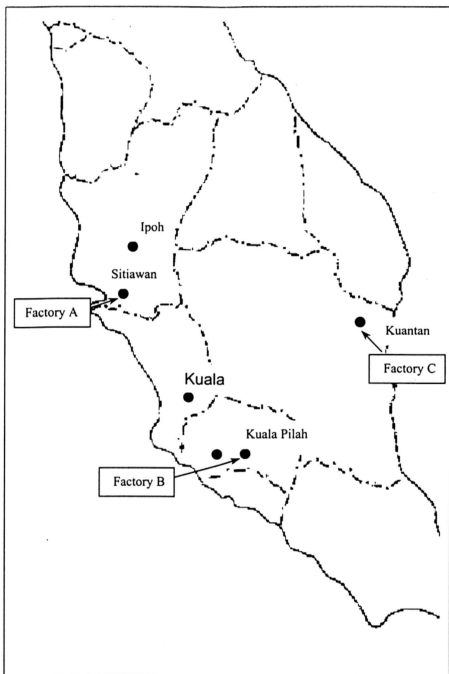
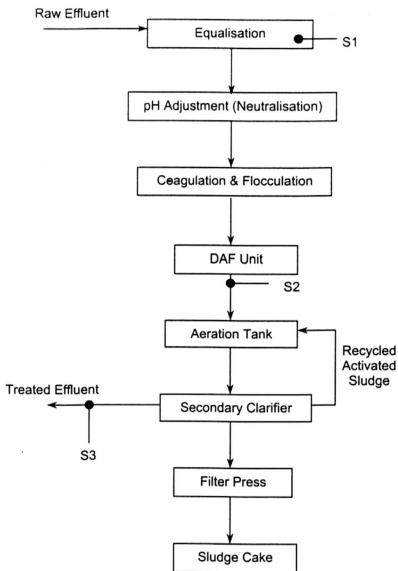


Figure 3.1 : Map of Peninsular Malaysia indicating Factory A, B and C



- S1 - Sampling point for raw effluent
- S2 - Sampling point for effluent after primary treatment (physical-chemical stage)
- S3 - Sampling point for treated effluent (after biological treatment)

Figure 3.2 : Schematic Flow Diagram of the WWTP indicating the Effluent Sampling Points

## 3.2 Chemical Analyses

Chemical analyses were conducted on the wastewater samples collected from Factory A, B and C. The three parameters investigated are BOD, COD and SS. These analyses were carried out in accordance to the American Public Health Association Standard Methods –1992 (APHA, 1992).

### 3.2.1 Biochemical Oxygen Demand (BOD)

BOD is an empirical determination of the amount of oxygen required to oxidise the organic matter in the sample. BOD is determined by incubating the water sample with aerobic microorganisms under specific conditions of time and temperature. The most widely used test, BOD<sub>5</sub> is based on a 5-day incubation period at 20°C. However, BOD<sub>5</sub> value does not represent the total BOD as the biological oxidation of organic matter takes a lot longer than 5 days to go to completion. Approximately 95-99% of the reaction is completed after 20 days, but as this is too long to wait for results, a 5-day incubation period is commonly adopted. For most domestic and industrial wastewater, the BOD<sub>5</sub> value represents between 60% and 80% of the total BOD (Radojevic and Bashkin, 1999).

The wastewater sample was first diluted with a suitable volume of oxygen-saturated water containing nutrients. It was recommended that dilution for raw sewage to be 30 to 100 times dilution (Radojevic and Bashkin, 1999). Three BOD<sub>5</sub> 250 ml bottles for each dilution of a sample were set up. One of the bottles was labelled as "Initial",

while the other two labelled as "Incubate". The dissolved oxygen (DO) in the "Initial" bottle was measured on Day one itself, and it was considered as initial DO. The two "Incubate" bottles containing the diluted wastewater samples were then placed in an incubator for 5 days under the environmental conditions of zero illumination, 100% humidity and at 20°C. The DO level in these two bottles was measured at the end of the 5-day incubation period. The average of the two was the final DO and was used in calculating the BOD<sub>5</sub> value. The difference in the two DO values and correction for dilution was equal to the amount of oxygen consumed, as indicated in the following equation:

$$\text{BOD} = (\text{Initial DO} - \text{Final DO}) \text{ mg/l} \dots\dots\dots\text{Equation 3.1}$$

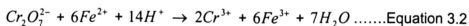
A blank using distilled water instead of the sample wastewater was also incubated alongside the samples. The DO value was determined by using YSI Model 57 DO meter, which was equipped with a BOD probe and a built-in stirrer.

### 3.2.2 Chemical Oxygen Demand (COD)

COD is a measure of the oxygen equivalent of the organic matter in a sample that is susceptible to oxidation by a strong oxidising agent. This test is widely used to measure the organic strength of domestic and industrial wastewater. COD can be related empirically to BOD, organic matter and organic carbon. The main difference between COD and BOD tests is that while BOD determines compounds that can be biologically oxidised, COD measures those substances which can be chemically

oxidised. As a rule of thumb, the BOD<sub>5</sub> values are normally 25 to 50% of the COD value on the same waste (Jackson, 1993). COD determination involves reacting the sample with an excess of an oxidising agent for a specified period of time, after which the concentration of unreacted oxidising agent is usually determined by a redox back-titration. The quantity of oxidising agent used up is determined by difference from the initial oxidant concentration.

Determination of COD was done by refluxing 20 ml of each sample and 30ml of distilled water to a final volume of 50 ml in a 500 ml refluxing flask. 25ml of the standard 0.00417M (equivalent to 0.25N) potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>) was added to the diluted samples prior to the reflux process, which lasted for 2 hours. Mercuric sulphate was added as the mercury would complex the halide interference prior to the addition of the potassium dichromate. Besides mercury sulphate, a mixture of strong sulphuric acid and silver sulphate (75ml) was added as a catalyst to speed up the oxidation process. The flask with all the relevant additives was then placed on the heating mantle and attached to condenser with running cooling water. A blank contained 50 ml of distilled water instead of sample wastewater was also prepared. After 2 hours of digestion, the unreacted dichromate, which remained in the solution, was titrated with ferrous ammonium sulphate containing orthophenanthroline plus ferrous iron complex (ferroin) as an indicator. The titration was stopped when the solution changed from blue-green to violet-red. The reaction is based on the following equation:



The amount of organic matter, which was oxidised, was in direct proportion to the volume of standard potassium dichromate consumed. The oxidisable organic matter is reported in terms of oxygen equivalents. Calculation of COD is as follows:

$$COD \left( \frac{mg O_2}{l} \right) = 8000 \times M \times \left( \frac{V_1 - V_2}{V_s} \right) \dots\dots\dots \text{Equation 3.3}$$

where

- $V_1$  = volume of ferrous ammonium sulphate titrant used to titrate the blank (ml)
- $V_2$  = volume of ferrous ammonium sulphate titrant used to titrate the sample (ml)
- $V_s$  = volume of sample (ml), and
- $M$  = exact concentration of ferrous ammonium sulphate titrant (mol/l) as determined in the standardisation titration above.

### 3.2.3 Suspended Solids (SS)

The term 'solids' refers to the quantity of solid matter remaining in a water sample after drying or igniting at a specified temperature. There are several categories of solids can be defined: total (TS), dissolved (DS), suspended (SS), settleable (SETS), fixed and volatile. The dissolved and suspended solids in water are differentiated by means of filtration. The procedure for measuring SS is a simple gravimetric analysis involving the difference in weights before and after a sample of water is passed

through a glass fiber filter. The filter with the retained residue was then dried in a convection oven at 103 to 105°C until a constant weight was obtained.

Prior to the filtration process, the filter was washed in a filter holder under suction with successive small volumes of laboratory water. The filter paper was then removed from the holder and dried in oven at 105°C for 1 hour. After the drying step, it was cooled in a desiccator and subsequently weighed. The drying, desiccating and weighing steps were repeated until a constant weight was achieved. The filter paper was placed back into the filter assembly. The sample wastewater was filtered through the filter under slight suction. Once all the sample wastewater had gone through the filter, the filter was removed and dry in oven at 105°C for 1 hour. After the drying, it was cooled in a desiccator and weighed. The SS was calculated as follows:

$$SS \left( \frac{mg}{l} \right) = 1000 \times \frac{(M_r - M_b)}{V} \dots\dots\dots \text{Equation 3.4}$$

where

- $M_r$  = weight of the filter paper after sample filtration (mg)
- $M_b$  = weight of the filter paper prior to sample filtration (mg), and
- $V$  = volume of the sample (ml)