# CHAPTER 3: MATERIALS AND METHODS

# 3.0 DESCRIPTION OF EXISTING WASTE WATER TREATMENT PLANT

The current waste water treatment plant provides physical and chemical treatment for waste water generated from the production wash-up process. The treatment plant consist of an equalisation basin, a primary settling tank, a flash mixing tank (where sulphuric acid, lime, alum, polymers and bio-nutrients are added for pH control and coagulation), two aeration tanks, a secondary settling tank and two holding tanks.

Waste water from the manufacturing plant is conveyed by gravity flow to the physical-chemical treatment plant. Here the colloidal particles or suspended solids in the waste water, coloured materials, BOD and COD as well as metals are reduced to levels within Standard B limits of the Malaysian Environmental Quality Act 1974 (Amendment 1996) prior to being discharged to a public sewer system.

The present study focuses on tertiary treatment of the effluent via chemical coagulation processes combined with cross-flow microfiltration as the polishing treatment to produce water, which is of sufficient quality for reuse within the manufacturing facility.

### 3.1 EXPERIMENTAL METHODS

# 3.1.1 Sampling Procedures

Composite samples were collected from two points, the equalisation tank and the effluent discharge out fall. Sampling was conducted over a period of 8 months. A total

of 20 samples were collected from each point. Sampling of the waste water was carried out according to Standard Methods for the Examination of Waste Water published by the American Public Health Association, 19th Edition 1997.

#### 3.1.2 Waste Water Characterisation

Four samples were analysed, raw waste water from the equalisation tank, treated effluent emanating from the out fall, treated effluent after undergoing tertiary treatment (chemical-coagulation) and effluent samples after undergoing tertiary treatment and membrane filtration. All samples were analysed following the procedures outlined in the Standard Methods (APHA), 1997.

# 3.1.3 Chemical Coagulation and Analysis

Laboratory scale evaluation of chemical coagulation and flocculation was performed using a six-place jar test apparatus equipped with multi-speed mixing stirrers. The jar test procedure included 5 minutes high shear mixing at 100 rpm, followed by 30 minutes flocculation at 20 rpm and a 1 hour settling sequence. Subsequently, a known volume of supernatant was withdrawn using a hypodermic needle from 1.6 cm below the surface.

The suspended solids concentration (T), was determined by turbidity meter (Jenway, UK Model No.6035). The relative turbidity, T/T<sub>0</sub> where T<sub>0</sub> is T in the absence of chemical coagulants was employed in order to assess the efficiency of the coagulant and flocculant used. The pH was measured with a Hanna meter (model 8417) which used a glass/calomel combination electrode that was standardised with appropriate

buffer solution. Redox potential measurements were recorded using a silver/silver chloride electrode model 320 from Corning. The inorganic chemicals like alum (Fluka Chemicals), calcium hydroxide (Ajax Chemicals) and anionic polyelectrolyte (Aldrich Chemicals) used for flocculation were of Analar grade and commercially available. Polyelectrolyte (anionic polyacrylamid) was added to the sample as a freshly made up, 0.25 % solution. Fresh polyelectrolyte solution was prepared daily from a 3 % stock solution.

Initially, the optimum pH for the function of alum and lime were determined. Subsequently varying doses of alum were applied. Varying doses of polyelectrolyte were then added at the optimum pH and alum dose. The mixing time, mixing speed, age of polymer, and mixer geometry were held constant to avoid the introduction of additional variables to the system. The removal efficiencies were measured in terms of turbidity and Chemical Oxygen Demand (COD). Once the optimum dosage of alum and polyelectrolyte had been determined, larger quantities of waste water was treated and a full analysis of parameters listed in Standard B of the Industrial Effluent Regulation 1989 was carried out. All parameters were analysed according to procedures outlined in Standard Methods (APHA 1997).

#### 3.1.4 Membrane Filtration

A bench scale cross-flow microfiltration membrane unit (Sartorius GmbH Model No. SM 16650) was employed to accomplish better clarification and disinfection of the treated effluent for recycle purposes. The membrane had a pore size of  $0.2 \, \mu m$ , an effective area of  $0.08 \, m^2$  and a transmembrane pressure of  $0.3 \, bar$ .

The set-up consisted of a feed tank, a pump with adjustable speed and the filter equipment. The trial was carried out utilising 10 litres of treated effluent and the permeate was collected in a measuring cylinder. The cumulative volume of the permeate was recorded at every 15 second interval.

#### 3.1.5 Microbial Tests

The effluent emanating from the waste water treatment plant (WWTP) and treated effluent before and after undergoing membrane filtration were tested for microbiological contamination. The samples were collected in sterilised bottles and stored at 4°C prior to the tests which were conducted within 24 hours of sample collection. pH measurements and redox potential measurements of the stored samples were then recorded. The samples were then screened for microbiological contamination. Growth of microorganisms was assessed after a 48 hours incubation period. Total viable counts were conducted for samples showing positive results at the screening stage.

## 3.1.5.1 Redox Potential and pH Measurements

pH measurements were recorded at ambient temperatures using a pH meter with a combination electrode. Redox potential measurements were recorded using a silver/silver chloride electrode.

## 3.1.5.2 Screening For Aerobic Microbiological Contamination

Aliquots of each sample were streak inoculated onto plates of nutrient and malt extract agars for the detection of bacteria and fungi (including yeast) respectively. After incubation for at least 48 hours at 25°C and /or 30°C, the resulting microbiological growth was visually assessed using a rating scale of 0 - 6 with 0 indicating no growth and 6 indicating dense growth. This visual assessement is just to indicate whether the samples were contaminated. Quantitative assessment will be conducted for samples that show positive microbial growth.

# 3.1.5.3 Screening For Anaerobic Sulphate Reducing Bacteria

Each sample was streak inoculated into tubes of SIM (sulphate iron media) / Iron Sulphate Agar. After incubation at 30°C, the resultant growth of sulphate reducing bacteria was visually assessed according to the degree of black coloration developing.

## 3.1.5.4 Total Viable Count by Miles and Misra Technique

Serial dilutions of each sample were prepared in sterile 1/4 strength Ringer's Solution. Duplicate 25 µl drops of each dilution were pipetted onto the surface of well dried nutrient and malt extract agars for the detection of bacteria and fungi (including yeast) respectively. After incubation at 25°C and/or 30°C for a minimum of 24 hours (bacteria) and 48 hours (moulds and yeast), the number of colonies from a "countable" drop were recorded.

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