# CHAPTER 3

# METHODOLOGY

Alternative chemicals tested in this study are soda ash, caustic soda and potassium hydroxide. Performance of the alternative chemicals in the treatment of wastewater was conducted at laboratory scale using jar tests. Sample used was wastewater from Sultan Azlan Shah Power Station. Chromium reduction stage is the first stage in the wastewater treatment as described in chapter 1. After this chromium reduction stage the alternative chemicals were used in place of hydrated lime for pH adjustment.

## 3.1 Experimental Design

The experiment conducted was adopted from the method used by the contractor during commission of the wastewater treatment plant. The method is based on ASTM D 2035 – 80 with some changes made to simulate the conditions of the main wastewater treatment plant.

800 ml of wastewater sample was poured into each of the four beakers and was placed in a jar test equipment. The speed of the stirrer was set at 200 rpm. The pH was dropped to about 3.0 to simulate the chromium reduction stage using 5 % hydrochloric acid with the aid of a calibrated pH meter. Since the chromium content was very low the ferrous sulphate was not added.

After the chromium reduction stage the pH of the contents in the beakers were raised to 7.5 using hydrated lime, potassium hydroxide, soda ash and sodium hydroxide respectively. The calibrated pH meter was also used for this step. Polyaluminium chloride(PAC) equivalent to 10.7 ppm was then added into each of the beakers. After 15 minutes the floc size was noted. Anionic polyelectrolyte(PE) equivalent to 2.5 ppm was added into each of the beakers and the stirring was continued for 5 minutes. The stirrer speed was then reduced to 40 rpm and stirred for 30 minutes. At the end of 30 minutes the stirrer was stopped and the time taken for the visible floc to settle to the bottom of the beaker was noted. The floc size was also noted.

The supernatant solution of each of the beakers was taken and analysed for :

a) Final pH after flocculation and settling.
b) Chemical Oxygen Demand
c) Suspended Solids
d) Chromium Hexavalent
e) Copper
f) Manganese
g) Zinc
h) Boron
i) Iron
j) Oil & Grease
k) Turbidity

Above jar tests were repeated for pH values of 8.0, 8.5, 9.0, 9.5 and 10.0 after chromium reduction stage. Sludge generated at each pH for each of the chemical was measured using gravimetric method. Main criteria used to judge the performance of the alternative chemicals was settling time of floc and floc size. The apparatus used and procedure of the test is attached as appendix 1.

Replicate test was not carried out due to the broad pH range and many parameters that were tested for supernatant of each pH.

## 3.2 Wastewater Sample

20 liters of wastewater sample was collected from the primary holding pond of the main wastewater treatment plant. The sample was collected in plastic containers and was properly capped. This sample was used to conduct jar test using hydrated lime and the other chemicals at various pH. This is to ensure that test is carried out on same sample so that the results can be comparable.

The wastewater sample was also tested for various parameters mentioned for supernatant solution after jar test.

## 3.3 Chemical Analysis

Jar tests were carried out on same effluent sample while varying the pH and chemicals used for pH adjustment and after chromium reduction stage. The effluent sample was collected in early September 2007. Analysis of supernatant solution resulting from the jar test was carried.

### 3.3.1 pH

pH expresses the hydrogen ion concentration of a solution. It is an important parameter for wastewater treatment as certain reactions are pH dependent for optimal performance. There is range of pH limit for discharge of effluents.

The pH of a solution is expressed as the negative logarithm of the hydrogen ion concentration as per the following equation.

$$pH = -\log_{10} [H^+]$$

The pH of the wastewater sample and the supernatant solutions were determined using a standard portable pH meter at room temperature. The pH adjustment after chromium reduction stage for jar test was also carried out with the use of the portable pH meter.

### 3.3.2 Chemical Oxygen Demand ( COD)

Metcalf & Eddy ( 1972) mentions that the chemical oxygen demand, (COD) , test is used to measure the content of organic matter of both wastewater and natural waters. The oxygen equivalent of the organic matter that can be oxidized is measured by using strong chemical oxidizing agent in an acidic medium. Potassium dichromate has been found to be excellent for this purpose. The test must be performed at elevated temperature. A catalyst ( silver sulfate ) is required to aid the oxidation of certain class of organic compounds.

The method used to test COD is method 8000 (HACH, 1997). In this procedure the samples are heated for two hours with strong oxidizing agent, potassium dichromate. Oxidizable organic compounds reduces the dichromate ion to green chromic ion (Cr (III) ). The digested sample is cooled and COD results are obtained using HACH DR/2010 spectrophotometer and its stored program.

### 3.3.3 Suspended Solids

By definition suspended solids is a total of the settleable solids and the filterable solids. This is true for the raw effluent sample. In the case of the supernatant solution taken after the jar tests, the settled solids were not included. The suspended solids tested for the supernatant is taken to indicate the suspended solids of sample after clarifiers, which will be minus the settled solids.

The method used for suspended solids is method 8006 ( HACH , 1997 ). In this method the suspended solids are determined by direct measurement using the stored program for the test in HACH DR/2010 spectrophotometer. This method is useful for quick checking of processes but is not specified by USEPA.

### 3.3.4 Chromium Hexavalent

The method used for determination of hexavalent chromium is method 8023 ( HACH , 1997 ). Hexavalent chromium is determined by the 1,5-diphenylcarbhydrazide method. The single dry power reagent contains an acidic buffer combined with 1,5-diphenylcarbhydrazide which reacts to give a purple color which is proportional to the amount of hexavalent chromium present. The hexavalent chromium concentration is obtained using HACH DR/2010 spectrophotometer and its stored program.

### 3.3.5 Copper

The method used for determination of copper is method 8023 ( HACH , 1997 ). Copper in the sample reacts with a salt of bicinchoninic acid contained in the copper reagent to form a purple colored complex in proportion to the copper concentration. The copper concentration is obtained using HACH DR/2010 spectrophotometer and its stored program.

### 3.3.6 Manganese

The method used for determination of manganese is method 8034 ( HACH , 1997 ). Manganese in the sample is oxidized to the purple permanganate state by sodium periodate, after buffering the sample with citrate. The purple color is directly proportional to the manganese concentration. The manganese concentration is obtained using HACH DR/2010 spectrophotometer and its stored program.

### 3.3.7 Zinc

The method used for determination of zinc is method 8009 ( HACH , 1997 ).

Zinc and other metals in the sample are complexed with cyanide. The addition of cyclohexanone causes a selective release of zinc. The zinc then reacts with the 2-carboxy-2-hydroxy-5-sulfoforamazyl benzene indicator. The zinc concentration is proportional to the resulting blue color. The zinc concentration is obtained using HACH DR/2010 spectrophotometer and its stored program.

### 3.3.8 Boron

Boron was tested using method 10061 ( HACH , 1997 ). Azomethine-H, a Schiff base, is formed by the condensation of an aminonapthol with an aldehyde by the catalytic action of boron. The boron concentration in the sample is proportional to the developed color. The boron concentration is obtained using HACH DR/2010 spectrophotometer and its stored program.

### 3.3.9 Iron

Iron was tested using method 8008 ( HACH , 1997 ). The reagent contains reducing agent to convert all iron to ferrous and 1,10-Phenanthroline indicator. Ferrous ion reacts with the indicator to form orange color which is proportional to iron concentration. The iron concentration is obtained using HACH DR/2010 spectrophotometer and its stored program.

### 3.3.10 Oil and Grease

Oil and grease was tested using EPA test method 413.2 . The test method uses solvent extraction and infrared spectroscopy to analyse. The oil and grease is extracted in the solvent H-997 and the oil and grease content is analysed using HOIRBA Oil Content Analyser OCMA-355.

### 3.3.11 Turbidity

Turbidity was tested using method 8195 ( HACH , 1997 ). The turbidity test measures the optical property of the water sample that results from the scattering and absorbing of light by particulate matter present. The nephelometric turbidity is obtained using HACH 2100N turbidimeter.

### 3.3.12 Floc Size

Floc size was determined based on the size of flocs produce at a particular stage. The sizes numbers used are as follows :

a) Number 3 for flocs sizes between 0.3 mm to 0.5 mm
b) Number 4 for flocs sizes between 0.5 mm to 0.75 mm
c) Number 5 for flocs sizes between 0.75 mm to 1.0 mm
d) Number 6 for flocs sizes between 1.0 mm to 1.5 mm
e) Number 7 for flocs sizes between 1.5 mm to 2.25 mm
f) Number 8 for flocs sizes between 2.25 mm to 3.0 mm
g) Number 9 for flocs sizes between 3.0 mm to 4.5 mm

A chart with different floc sizes was used to facilitate determination of floc size. Copy of the chart is attached as appendix 2.

### 3.3.13 Weight of Sludge Generated

The sludge was filtered using a pre-weighed filter paper. The filter paper together with the sludge was dried in an oven at 105 degrees Celsius until the weight was constant. The weight of filter paper and sludge after drying minus the weight of empty filter paper gives the weight of sludge generated.