

CHAPTER 3

EXPERIMENTAL STUDIES

3.1 Materials

The wastewater sample used for this work was obtained from the equalization basin of a soap and detergent manufacturing factory in the state of Selangor, Malaysia. Fresh samples were obtained daily to avoid deterioration of wastewater sample. The chemicals used were analytical reagent standard and are listed in **Table 3.1**.

Table 3.1 : List of Chemicals

Chemical	Company
Aluminum sulfate [$\text{Al}_2(\text{SO}_4)\cdot x\text{H}_2\text{O}$]	Ajax Chemicals, Australia
Ferric sulfate [$\text{Fe}_2(\text{SO}_4)\cdot 18\text{H}_2\text{O}$]	Ferak, Merck, Germany
Polyacrylamide	Aldrich Chemicals, USA
Sodium alginate	Fluka Chemica, Switzerland
Polyethylenimine	Sigma Chemicals, USA
Sodium hydroxide	Fluka Chemica, Switzerland
Sulfuric acid	Merck, Germany
Polyvinyl alcohol	Fluka Chemica, Switzerland
Granulated activated carbon	Fluka Chemica, Switzerland

3.2 Methods

3.2.1 Wastewater Characterization.

The characterization studies were carried out to determine the characteristics of the wastewater. Parameters of interest in this study were temperature, pH, suspended solids, zeta potential values, chemical oxygen demand and surface tension. All the measurements were done using Standard Methods (Standard Methods, 1995). The characteristics of the wastewater was determined in the following manner :

- a) The pH and temperature was measured using a Hanna pH meter model 8417.
- b) The zeta (ζ) potential was measured using a Zetasizer 4 from Malvern, UK.
- c) The chemical oxygen demand was measured using a test kit by Dr. Lange Co., Germany. In this method 0.2 ml of sample was put into a cuvette test (a test tube containing a mixture of sulfuric acid and potassium dichromate solution with silver sulfate as a catalyst). The mixture also contains mercury sulfate to mask chloride and then the cuvette was closed and inverted a few times to homogenize the mixture. The solution was heated for 2 hours at 148°C. It was then allowed to cool to room temperature before evaluation. It was evaluated by a spectrophotometer supplied by the test kit at a wavelength of 605 nm.

d) Suspended solids were measured by filtering a known volume of sample through a Whatman 42 filter paper that was preweighed and dried at 103°C for 1 hour and then cooled to room temperature in a dessicator. After cooling to room temperature, it was weighed again and the difference in weight was taken as the suspended solids value.

e) The surface tension reading were taken using Sensadyne model 6000 from Sendyne Instruments, USA. The measurements were carried out at 25°C and readings were taken in units of dynes/ cm.

3.2.2 Studies on Factors Influencing the Coagulation and Flocculation Process During Treatment of Wastewater.

In this study, the main focus was to determine the optimal condition for the coagulation and flocculation process in the treatment of wastewater. This was done by evaluating and determining selected important factors such as :

Coagulant Dosage	0.5, 1.0, 1.5	0.5, 1.0, 1.5	0.1, 0.2, 0.3	0.1, 0.2, 0.3	0.1, 0.2, 0.3
Coagulant Dosage	(a) Type and optimum coagulant dosage.				
Mixing Time	(b) The optimum pH.				
Mixing Intensity	(c) The optimum mixing time.				
Settling Time	(d) The optimum mixing intensity.				
Settling Intensity	(e) The optimum setting time.				

In order to determine these factors in the coagulation and flocculation process, the jar test was applied. The jar test was conducted under actual plant operating conditions. The equipment used in this work was a Chemix Flocc - Tester model CL4,

Malaysia. The set consist of a four-paddle stirrer rig. The stirrers were equipped with variable speed controls.

3.2.2.1 Determination of the Type of Coagulant and Optimal Coagulant Dosage

The two main types of coagulant agent used were aluminum sulfate and ferric sulfate. Three types of coagulant aid (polyelectrolytes) were tested, and they were polyvinyl alcohol, sodium alginate and polyethylenimine. Duplicate sets of 500 ml of wastewater was subjected to the Jar Test with the conditions as stated in **Table 3.2**. The pH of the wastewater subjected to the following conditions was not adjusted.

Table 3.2 : Jar Test Conditions to Determine Type of Coagulant and Optimal Coagulant Dosage.

Coagulant	Aluminum sulfate	Ferric sulfate	Polyvinyl alcohol	Sodium alginate	Polyethylenimine
Coagulation Dosage (mg/l)	0.5, 1.0, 1.5,	0.5, 1.0, 1.5,	0.1, 0.2, 0.3,	0.1, 0.2, 0.3,	0.1, 0.2, 0.3,
Flash Mixing Stage at 100 rpm	60 seconds	60 seconds	30 seconds	30 seconds	30 seconds
Flocculation Stage at 30 rpm	30 minutes	30 minutes	30 minutes	30 minutes	30 minutes
Settling Stage at 0 rpm	30 minutes	30 minutes	30 minutes	30 minutes	30 minutes

After the settling stage, the sludge volume was determined. The supernatant of samples was taken without disturbing the sediment to analyze for zeta potential value and the percentage of turbidity removal.

3.2.2.2 Determination of Optimum pH

The effect of pH variation on the treatment condition was evaluated. The study focused on pH ranges from 3 to 12. Duplicate sets of 500 ml of wastewater was subjected to the Jar Test. The initial pH of the wastewater sample was adjusted by adding either sodium hydroxide (NaOH) or sulfuric acid to obtain the required pH. The optimal dosage of coagulation agent and coagulation aids was obtained from Section 3.2.2.1. The conditions for the flash mixing stage, the flocculation stage and the settling stage remained as stated in Table 3.2. After the settling stage, the supernatant of samples was taken without disturbing the sediment to analyze for zeta potential and the percentage of turbidity removal.

3.2.2.3 Determination of Optimum Mixing Time.

The effect of mixing time or reaction time at the flocculation stage on the treatment condition was evaluated. The study focused on mixing time ranging from 5 to 45 minutes at 30 rpm. Duplicate sets of 500 ml of wastewater were subjected to the Jar Test. The optimal dosage of coagulation agent and coagulation aids was obtained from Section 3.2.2.1 and the responding optimal pH was obtained from Section 3.2.2.2. The

conditions for the flash mixing stage and the settling stage remains as stated in **Table 3.2**. After the settling stage, the supernatant of samples was taken without disturbing the sediment. This was to analyze for zeta potential and the percentage of turbidity removal.

3.2.2.4 Determination of Optimum Mixing Intensity

The effect of mixing intensity at the flocculation stage on the treatment condition was evaluated. The study focused on mixing intensity ranging from 10 to 100 rpm. Duplicate sets of 500 ml of wastewater was subjected to the Jar Test. The optimal dosage of coagulation agent and coagulation aids was obtained from **Section 3.2.2.1**, the corresponding optimal pH was obtained from **Section 3.2.2.2** and the corresponding mixing time from **Section 3.2.2.3**. The conditions for the flash mixing stage and the settling stage remains as stated in **Table 3.2**. After the settling stage, the supernatant of samples was taken without disturbing the sediment to analyze for zeta potential and the percentage of turbidity removal.

3.2.2.5 Determination of Optimum Settling Time

The effect of settling time at the settling stage on the treatment condition was evaluated. The study focus on settling time ranging from 10 to 100 minutes. Duplicate sets of 500 ml of wastewater were subjected to the Jar Test. The optimal dosage of coagulation agent and coagulation aids was obtained from **Section 3.2.2.1**, the

corresponding optimal pH was obtained from Section 3.2.2.2, the corresponding mixing time for the flocculation stage from Section 3.2.2.3 and the corresponding mixing intensity for the flocculation stage from Section 3.2.2.4. The conditions for the flash mixing stage were as stated in Table 3.2. After the settling stage, the supernatant of samples was taken without disturbing the sediment to analyze for the zeta potential value and the percentage of turbidity removal.

3.2.2.6 Evaluation of Optimum Conditions

Duplicate sets of 500 ml of wastewater were subjected to the Jar Test with the optimal conditions obtained from Sections 3.2.2.1, 3.2.2.2, 3.2.2.3, 3.2.2.4 and 3.2.2.5. After the settling stage, the sludge volume and sludge weight were determined. The supernatant was collected to analyze for zeta potential value, surface tension, chemical oxygen demand and the percentage of turbidity removal.

3.2.2.7 Double Chemical Treatment.

The wastewater was treated with a coagulant, followed by a coagulation aid or polyelectrolyte in the stage flash mixing stage.

Duplicate sets of 500 ml of wastewater were subjected to the Jar Test. The most effective coagulant agent, with the optimal dosage, was added to the wastewater and subjected to 60 seconds of flash mixing at 100 rpm. Then the most preferred coagulant

aid was added and subjected to 60 seconds of flash mixing at 100 rpm. The most preferred coagulant and coagulant aid was determined from Section 3.2.2.1. The Jar test was carried out with the optimal conditions obtained for the most effective coagulation agent from Sections 3.2.2.2, 3.2.2.3, 3.2.2.4 and 3.2.2.5. After the settling stage, the sludge volume and the sludge weight were determined. The supernatant was collected to analyze for zeta potential value, surface tension, chemical oxygen demand and the percentage of turbidity removal.

3.2.3 Adsorption Studies

Granulated activated carbon was selected as the adsorption material. The material was dried at 100°C for 24 hours before use. In each set of experiment, duplicate sets of wastewater was allowed to react with the activated carbon in the following manner. Flash mixing at 100 rpm for 20 seconds and continued by slow mixing at 30 rpm for 10 minutes. The supernatant was filtered through Whatman 42 filter paper by a vacuum pump. The experiment was repeated with absorbent quantities ranging from 0.1, 0.2, 0.3 to 20 g maximum.

The surface tension and the color removal of the filtrate were determined. The surface tension readings were taken using Sensadyne model 6000 from Sendyne Instruments, USA. The measurements were carried out at 25°C and readings were taken in units of dynes/cm. Color removal was determined by using a spectrophotometer which recorded the absorbency of the filtrate at 384 nm and 392 nm. The spectrometer used was by C.K.B. Biochrom England, Model Novaspec II.