

# CHAPTER 3

## METHODOLOGY



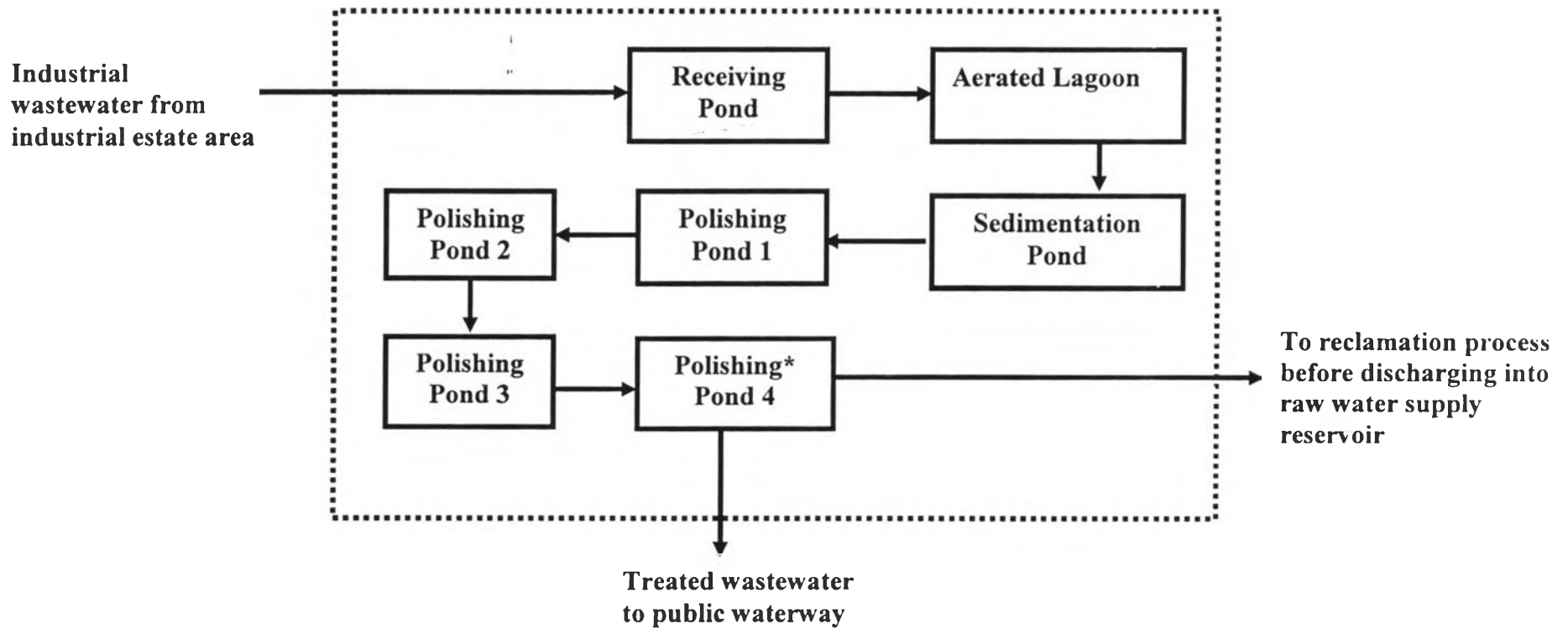
The study of reduction of trihalomethanes formation potential in reclaimed water from treated industrial estate wastewater was carried out in the Laboratory of Department of Environmental Engineering, Faculty of Engineering, Chiangmai University, Chiangmai, Thailand by using the raw water samples from the selected industrial estate located in northern Thailand over the entire period of study between October 2001 and February 2002. Descriptions of source of raw water sample, coagulation experiment, water sample and analytical parameter, analytical method and instrument were presented separately as follows:

### 3.1 Source of Raw Water Sample

Treated industrial wastewater in the final polishing pond of the central wastewater treatment plant of the Northern Region Industrial Estate in Lumphoon Province was considerably selected as raw water used in this study. Composited samples from five sampling points at peripheral area of the final polishing pond were collected in glass bottles with TFE-lined screw caps and preserved by sodium thiosulfate prior to storage at 4 °C. The pictorial view of the central wastewater treatment plant of the Northern Region Industrial Estate, Lumphoon Province and the diagram of wastewater treatment process are illustrated in Figure 3.1 and 3.2, respectively.



**Figure 3.1** Final polishing pond of the central wastewater treatment plant of the Northern Region Industrial Estate



\*Final polishing pond used as a source of raw water samples in this study.

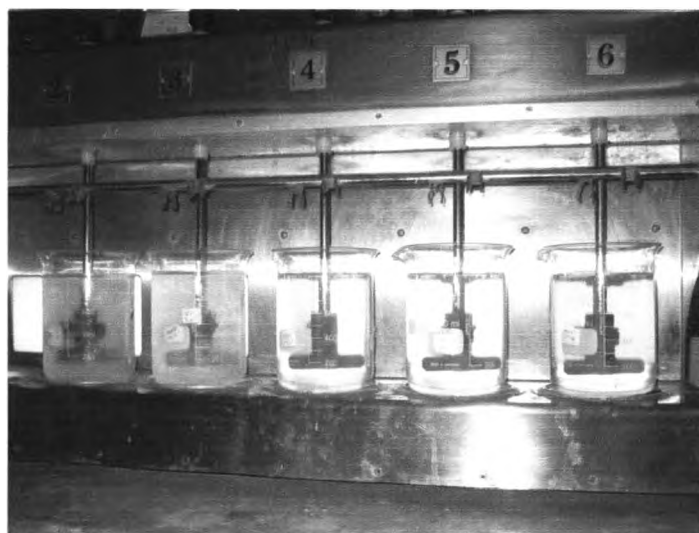
**Figure 3.2** Wastewater treatment diagram of the central wastewater treatment plant of the Northern Region Industrial Estate

## 3.2 Coagulation Experiment

### 3.2.1 Jar-Test Apparatus and Process

Since many factors influence coagulation and complex reaction involved, the coagulant dosage required for coagulation of particular water normally is determined from experiments. The experimental jar-test apparatus, as shown in Figure 3.3, was used for such a purpose in this study and the jar-test procedure is briefly described as follows.

Using a multistage stirrer apparatus, simultaneous tests were conducted on a series of samples covering a range of coagulant concentration. On the addition of the coagulant, the samples were rapidly mixed with 100 rpm. for 1 minute, followed by slow mixing with 30 rpm. for 30 minutes. The samples were then allowed to stand for 1 hour for settlement, after which turbidity, UV-254, TOC, DOC and SUVA were measured and the lowest coagulant dose giving adequate removal noted. Using the same concentration of coagulant, second similar sets of tests were also performed on pH-adjusted samples so as to determine the optimum pH values for such coagulation.



Jar-test apparatus (Miyamoto Riken, Model JMD-6)

**Figure 3.3** The experimental jar-test apparatus used in this study

### 3.2.2 Jar-Test Condition

The jar-test procedure as stated previously were experimented under the conditions of coagulation as depicted in Table 3.1

**Table 3.1** The experimental jar-test condition

Coagulant	Coagulant dosage (mg/L)	pH adjustment
Alum	0, 10, 20, 40, 60, 80	Uncontrolled pH and controlled pH at 6.5, 6 and 5.5
Ferric Chloride	0, 10, 20, 40, 60, 80	Uncontrolled pH and controlled pH at 6.5, 5.5 and 5

### 3.2.3 Alum

Regular-grade alum ( $\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$ ) typically in powder form containing approximately 97 percent of  $\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$  was used for preparing the 20 mg/mL stock solution.

### 3.2.4 Ferric Chloride

Analytical-grade ferric chloride ( $\text{FeCl}_3$ ) available as powder containing approximately 99 percent of  $\text{FeCl}_3$  was used for providing the 20 mg/mL stock solution.

## 3.3 Water Sample and Analytical Parameter

There are a number of parameters used to express the THMFP and performance of coagulation process for THMFP reduction as well as the correlation of surrogate parameters for NOM. Sample (Raw water, Treated Industrial Estate Wastewater), sample in sedimentation after jar-test experiment and sample in sedimentation after jar-test experiment and 7 days holding were considerably collected as the water samples for analysis. The water samples and analytical parameters performed during the experiments were distinguishably presented as shown in Table 3.2 and also conclusively in term of schematic diagram as illustrated in Figure 3.4

Table 3.2 Water sample and analytical parameter

Parameter	Sample before jar-test experiment (Raw water, Treated Industrial Estate Wastewater)	Sample in sedimentation after jar-test experiment	Sample in sedimentation after jar-test experiment and 7 days holding
pH	√	√*	√*
Turbidity (NTU)	√	√**	-
Alkalinity (mg/L as CaCO <sub>3</sub> )	√	√**	-
UV-254 (1/cm)	√*	√*	-
TOC (mg/L)	√*	√*	-
DOC(mg/L)	√*	√*	-
SUVA (L/mg-m)	√*	√*	
Temperature (°C)	√	√*	√*
Free chlorine residual (mg/L)	√	-	√*
TTHM <sub>0</sub> (μg/L)	√	√*	-
TTHM <sub>7</sub> (μg/L)	√	-	√*

$$\text{THMFP} = \text{TTHM}_7 - \text{TTHM}_0$$

\*Filtered samples by using 1.2 μm GFC

\*\*Both unfiltered sample (supernatant) and filtered sample

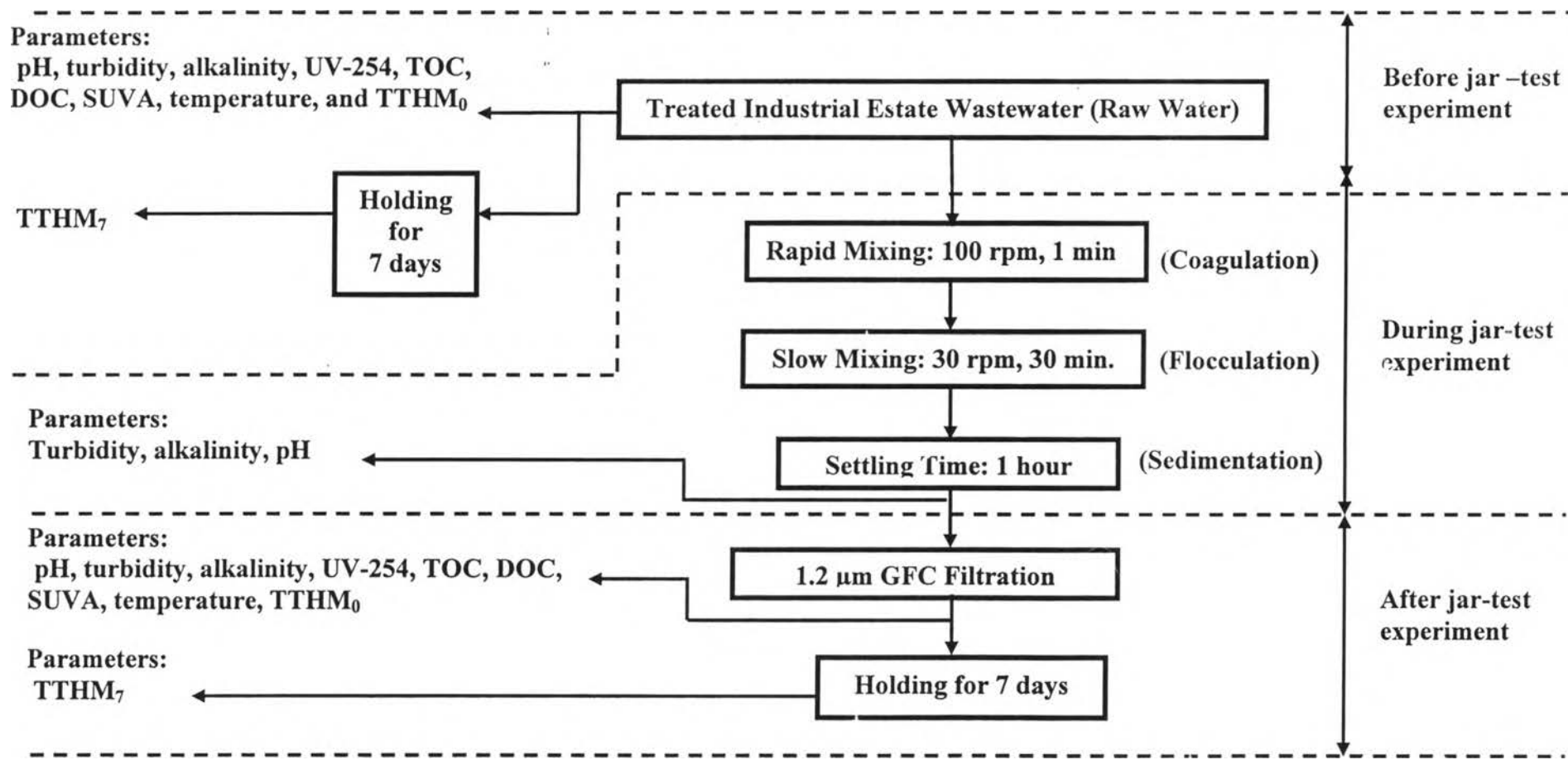


Figure 3.4 Water sample and analytical parameter in this study

### 3.4 Analytical Method and Instrument

**Alkalinity** was determined in accordance with standard method 2320, section 2320 B, Titration Method.

**Turbidity**, Direct measurement by using HACH 2100, Turbidity meter

**pH** was directly measured by Horiba pH meter model D-13E with an accuracy of  $\pm 0.01$  pH unit. The unit was calibrated daily with buffer solution at pH 4.00 and 7.00

**UV 254** was measured in accordance with standard method 5910 B, Ultraviolet Absorption Method. The samples were filtered through a prewashed 0.45  $\mu\text{m}$  cellulose acetate membrane prior to measurement.

**TOC and DOC** were analyzed in accordance with standard method 5310, Total Organic Carbon (TOC) and section 5310 C, Persulfate-Ultraviolet Oxidation Method. Water samples were filtered through glass-fiber filter of nominal pore size (1.2  $\mu\text{m}$ ) prior to TOC analysis. Water samples were filtered through a prewashed 0.45  $\mu\text{m}$  cellulose acetate membrane prior to analyzing DOC

**Trihalomethanes**, The THMs species such as chloroform, dichlorobromoform, dibromochloroform, and bromoform were determined. Three analytical methods were used to analyze THMs, using bromofluorobenzene standard solution as internal standard, as briefly described below:

- **First Method**, THMs were measured in accordance with standard method 5710, Formation of Trihalomethanes and Other Disinfection By-Products. Gas Chromatography was used to measure THMs under the condition as follow:

*Inlet Condition*

Mode: Split, Initial temp: 225°C., Pressure: 31.33 psi,  
Split ratio: 10:1S split flow 15.9 mL/min, Gas Type: Helium and Total  
flow: 20.5 mL/min

*Oven Condition*

The condition of temperature programs of oven adjusted for analyzing THMs are shown in Table 3.2:

**Table 3.3** Temperature program for analyzing THMs

Ramp	Rate (°C/min)	Final temp. (°C)	Holding time of final temp (minute)	Remark
1	15	100	1.00	Initial temp.: 75°C, Initial temp. Holding Time 1.00 min
2	15	130	1.00	-
3	15	180	1.00	-

*Detector Condition*

Temperature: 300 °C, Mode: Constant make up flow, Makeup flow: 60 mL/min, Makeup Gas Type: Nitrogen

- **Second method**, Free Chlorine Residual

Free chlorine residual was measured in accordance with Standard method 4500-Cl G. DPD Colorimetric Method.

- **Third method**, Liquid-Liquid Extraction

Water samples were extracted in accordance with standard method 6232B Liquid-Liquid Extraction Gas Chromatography Method

The summary of analytical methods and instruments used in this study were depicted in Table 3.3



**Table 3.3** Analytical method and instrument

Parameter	Analytical method	Standard	Analytical Instrument
pH	Direct measurement	-	Horiba pH-meter, Model D-13E
Turbidity	Direct measurement	-	HACH, 2100 Turbidity Meter
Alkalinity	Titration Method	Standard method 2320B	-
UV-254	Ultraviolet Absorption Method	Standard method 5910 B	Perkin-Elmer Model Lambda 25, UV/VIS spectrometer
TOC	Persulfate-Ultraviolet Oxidation Method	Standard method 5310C	O.I. analytical 1010 TOC Analyzer
DOC	Persulfate-Ultraviolet Oxidation Method	Standard method 5310C	O.I. analytical 1010 TOC Analyzer
Temperature	Direct measurement	-	Horiba Thermometer, Model D-13E
Free chlorine residual	Colorimetric Method	Standard method 4500-Cl G	Perkin-Elmer Model Lambda 25, UV/VIS spectrometer
TTHM <sub>0</sub>	Formation of Trihalomethane and Other Disinfection By-Products and Liquid-Liquid Extraction Gas Chromatography Method	Standard method 5710 and 6232B	Agilent 6890 Series Gas Chromatography with ECD detector
TTHM <sub>7</sub>	Formation of Trihalomethane and Other Disinfection By-Products and Liquid-Liquid Extraction Gas Chromatography Method	Standard method 5710 and 6232B	Agilent 6890 Series Gas Chromatography with ECD detector