CHAPTER 3
MATERIALS AND METHODS

3.1 Materials and Equipment

3.1.1 Wastewater

Wastewater used in this study was a real septic tank effluent collected from the outlet of septic tanks located at Chia Nan University of Pharmacy and Science, Taiwan. The effluent collection point is illustrated in Figure 3.1. The characteristics of septic tank effluent are shown in Table 3.1.

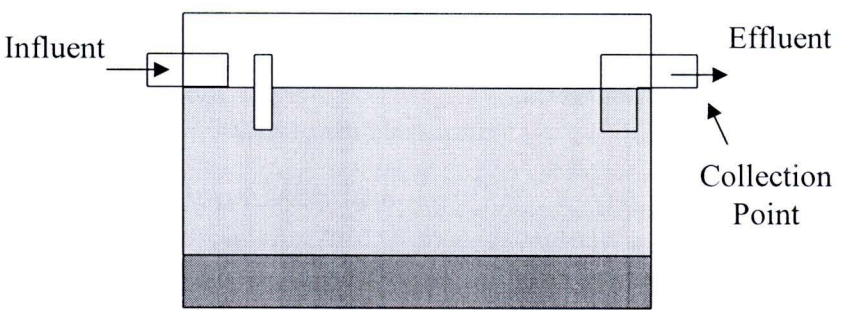

Figure 3.1 Septic tank effluent collection point.

Table 3.1 Septic tank effluent characteristics

Parameter*	Range
DO	0 - 1.77
pH	7.5 - 8.5
TCOD	100 - 279
SCOD	29 - 200
TN	52 - 932
NH_4-N	30 - 165
NO_2-N	ND - 1.1
NO_3-N	ND - 10.5

*all concentrations are in mg/L except pH, ND: not detected

3.1.2 Membrane bioreactor

The membrane bioreactor was designed by incorporating an in-line flocculation unit with a hollow fiber type aerobic membrane bioreactor as shown in Figure 3.2. Some important membrane module characteristics are shown in Table 3.2.

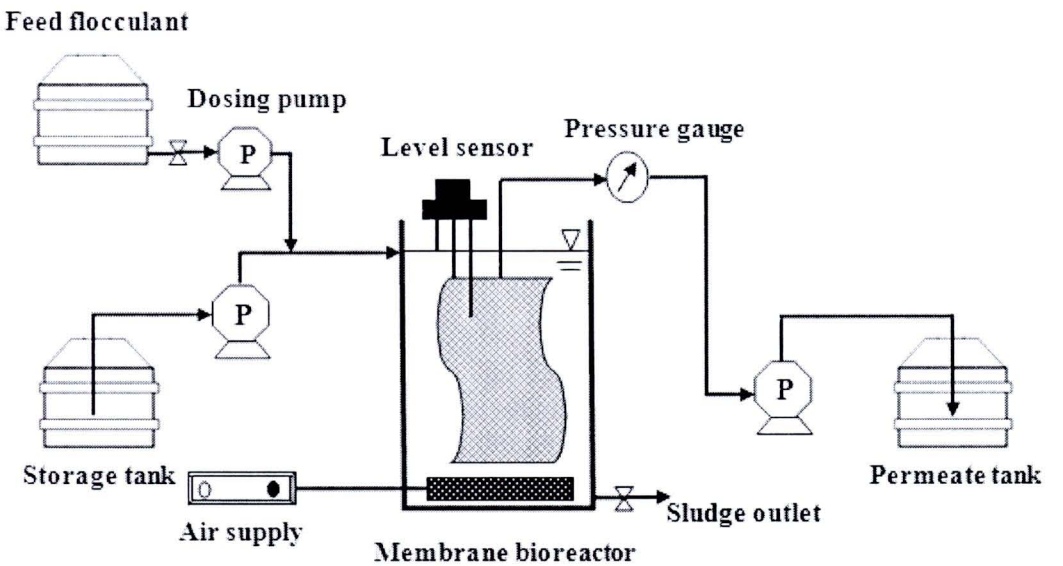

Figure 3.2 Schematic diagram of MBR tank.

The system consisted of five main components as follows

1) The reactor: A 12-litre rectangular tank made of clear acrylic material, housing a PVDF hollow fiber membrane. The mean pore size of the membrane is 0.1 μm.
2) Dosing system: An automatic flocculant feed pump was used to inject flocculants into the wastewater feed line through a tee fitting.
3) Feed and permeate pumps: Two rotary pumps with a suitable range of flow rate.
4) Air supply: An air compressor used to provide aeration through a soaker hose placed at the bottom of the reactor.
5) Storage and permeate tank: A container of the system influent and effluent for further analysis.

Table 3.2 Hollow fiber membrane module characteristics

Material	Polyvinylidencefluoride (PVDF)
Pore size	0.1 μm
Length	15 cm
Diameter	0.54 mm
Total surface Area	0.120 m^2

3.1.3 Flocculants

Since this study focused on the effects of different flocculant on the system performance, hence the PAM with different kind of charges including cationic-PAM, anionic-PAM and nonionic-PAM were selected for this study. All PAMs used in the evaluations were received in granular form. Stock solutions containing PAM (0.1% w/w) were made by slowly adding the PAM to a flask of stirring, distilled water and mixing for at least 24 hours at room temperature.

3.2 Experimental Procedure

The experimental procedures are shown in the following steps and conclusively described in the diagram shown in Figure 3-3.

1. The system was started-up and operated without flocculant addition until reaching the steady state. The feed and effluents were regularly sampled and analyzed for parameters which are shown in Table 3.3.
2. The mixing liquid in MBR tank and the raw septic tank effluent were taken 200 mL for jar test experiment with three different PAM flocculants under 10 levels of dosage (0.1, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, 5.0 and 10.0 mg/L). The results of the jar test were used to determine the flocculant and dosage that were employed in the subsequent in-line flocculation MBR system.
3. The appropriate type and dosage of flocculant that obtained from the jar test was continuously added into the system with wastewater feeding. The system was operated to reach the steady state and then change to next dosage for further study.

Table 3.3 Parameters and Analytical Methods

Parameter	Frequency	Method*
pH	Daily	pH Meter (YSI Environmental pH 100)
Temperature	Daily	Thermometer
Turbidity	Daily	Turbidity Meter (Hach 2000)
TCOD, mg/L	Three times per week	Close Reflux Method
SCOD, mg/L	Three times per week	Close Reflux Method
MLSS, mg/L	Three times per week	Filtration, Evaporation
MLVSS, mg/L	Three times per week	Filtration,Volatilization
TKN, mg/L	Three times per week	Kjedahl Method
NO_3^-, NO_2^-, mg/L	Three times per week	UV Spectrophotometric Method

*Standard Method for the Examination of Water and Wastewater (APHA, AWWA, WPCF, 1992

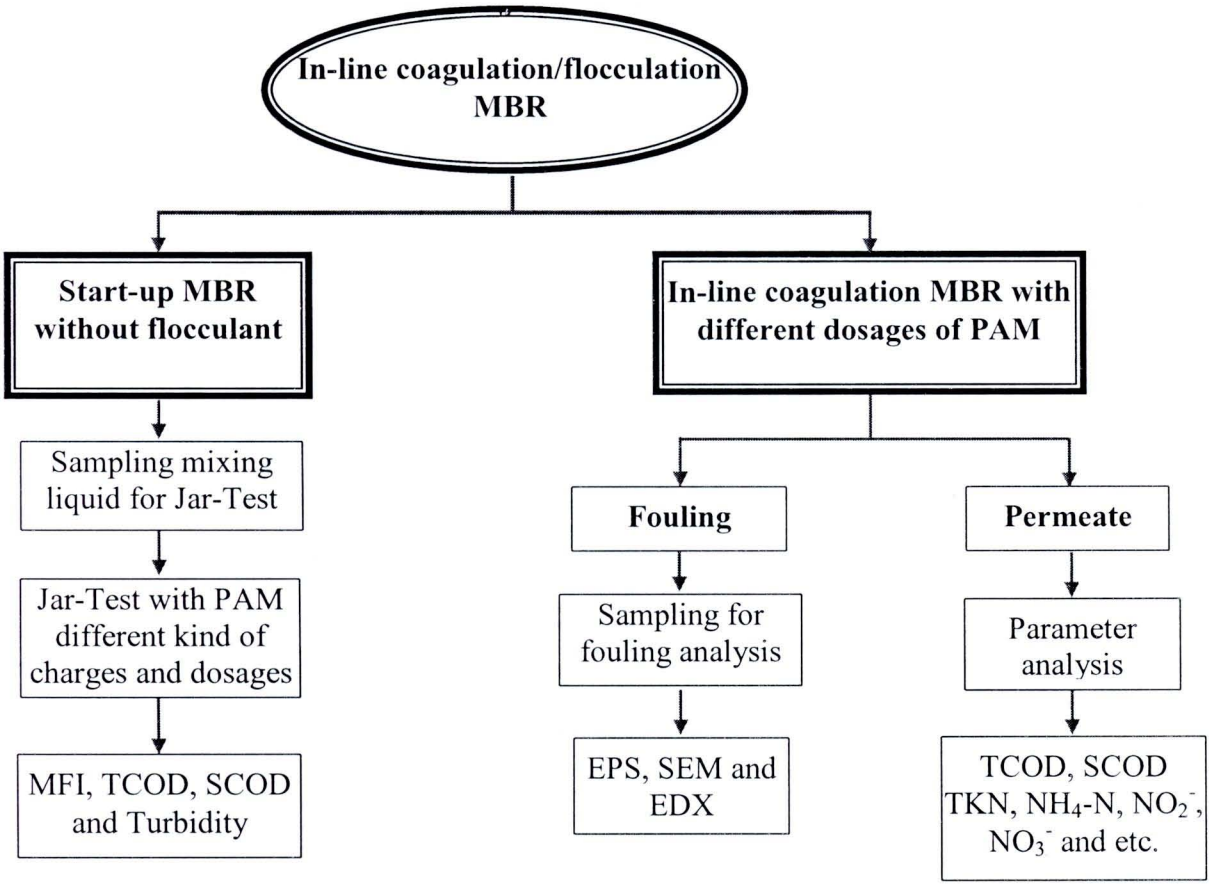

Figure 3.3 Diagram of experimental procedure.

3.3 Jar Test Experiment

The jar tests indicated the performance of each flocculant with respect to residual turbidity, TCOD, and SCOD removal to determine the flocculant doses that were employed in the subsequent in-line flocculation membrane bioreactor system. On the other hand, in order to determine the effect of flocculation on membrane fouling potential in MBR system, jar tests were also employed as a pre-procedure of MFI study.

Jar tests were carried out using a magnetic stirrer with rpm controlled function (Fargo MS-203 stirrer). In each test, 200 mL of sample was placed in the container and predetermined dosage of flocculant stock solution was added. The mixing protocol employed was rapid mixing for 2 min at 150 rpm followed by slow mixing for 15 min at 30 rpm, and eventually followed by settling for 30 min. After settling, the supernatant was collected for turbidity, TCOD and SCOD measurements. Figure 3.4 illustrates a jar test unit.

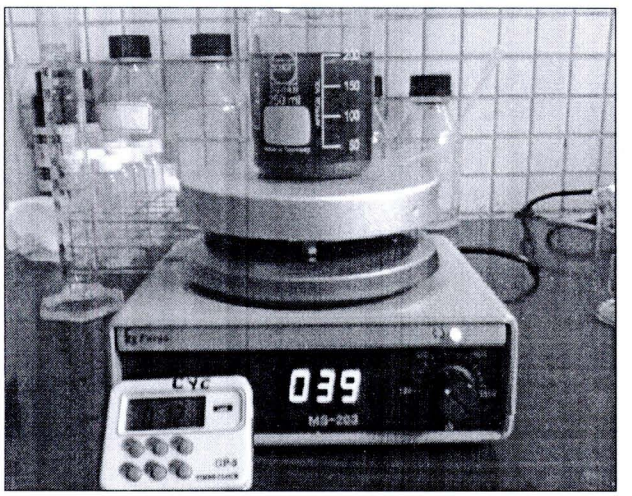

Figure 3.4 Magnetic stirrer (Fargo MS-203 stirrer).

3.4 Fouling Index Measurement

The MFI of mixing liquid from MBR tank was determined with a test membrane (pore size 0.1 and 0.45 μm) placed in a dead-end filter holder (membrane cell). The feed was conveyed from a 2.5 L vessel to the membrane at constant pressure (2 bars) provided from compressed air and measured by a pressure gauge. Permeate flow was measured with an analytical balance (AND GF6000) which is connected to the computer (PC) for data collection. Index values were determined from post-processed data, and corrected to reference conditions. Figure 3.6 illustrates the MFI equipment.

This index is based on cake filtration for constant pressure where the increase of total resistance is attributed to cake formation on the membrane surface (Equation 3.1) (Javeed *et al.*, 2009).

$$\frac{t}{V} = \frac{\eta \cdot R_m}{\Delta P \cdot A} + \frac{\eta \cdot \alpha \cdot c_v}{2 \cdot \Delta P \cdot A^2} \cdot V \qquad \textbf{(3.1)}$$

With

$$a = \frac{\eta \cdot R_m}{\Delta P \cdot A} \quad , \quad MFI = \frac{\eta \cdot \alpha \cdot c_v}{2 \cdot \Delta P \cdot A^2}$$

Equation 3-1 can be simplified to

$$\frac{t}{V} = a + MFI \cdot V \qquad \textbf{(3.2)}$$

with membrane surface area, A, particle concentration, c_v, in feed water, particle diameter, d_p, applied transmembrane pressure, ΔP, flow rate, Q, membrane resistance,

R_m, filtration time, t , filtrate volume, V, average specific cake resistance, α, and water viscosity, η.

The MFI is the value of the gradient of the linear section by plotting *t/V* versus *V*. This relationship can be presented graphically as in Figure 3.5.

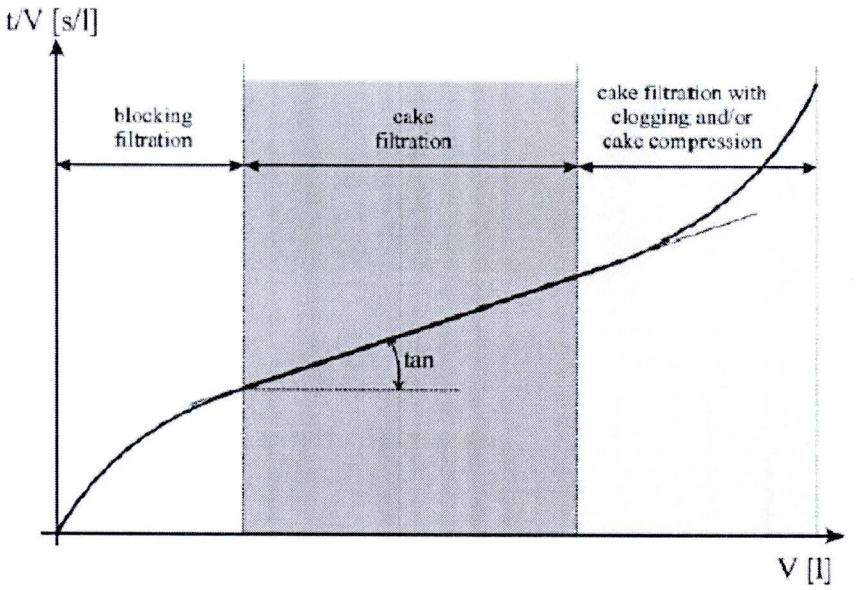

Figure 3.5 Cake filtration curve.

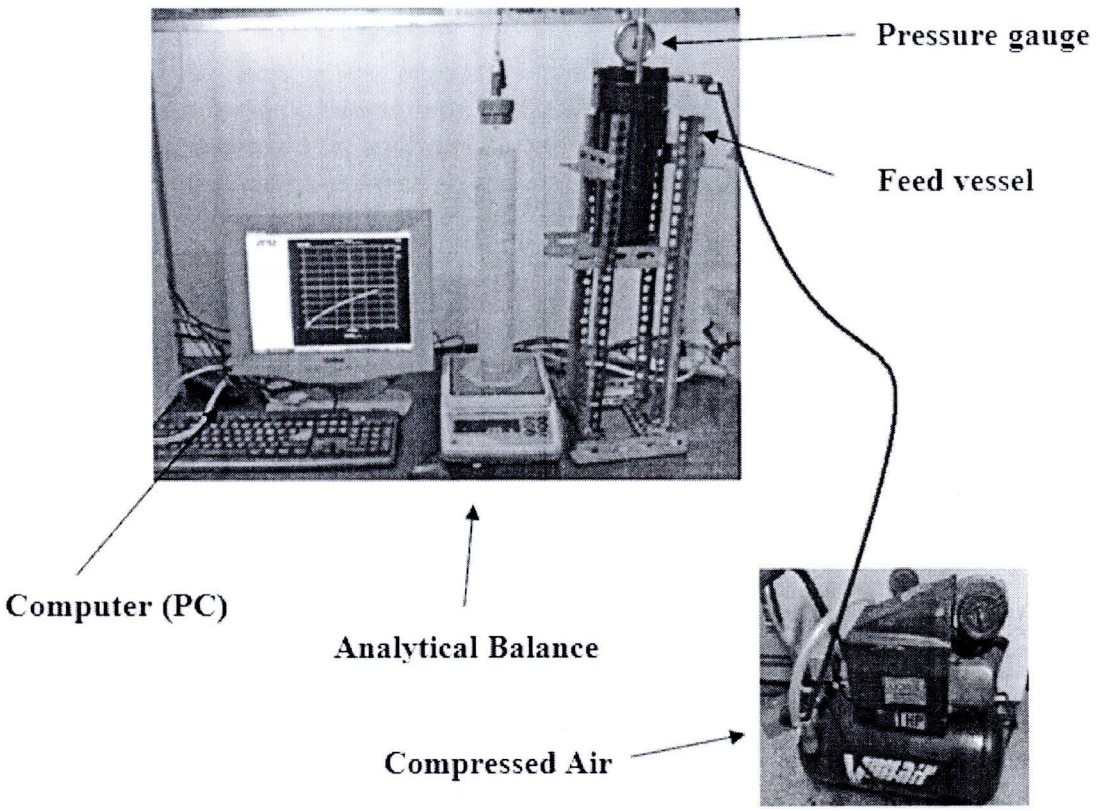

Figure 3.6 MFI equipment.

3.5 Membrane Fouling Study

The membrane fouling rate was determined in terms of transmembrane pressure which can be measured using pressure gauges. Foulants at the membrane surface were characterized by scanning electron microscope (SEM) and energy dispersive X-ray analyzer (EDX). The fouled membrane was collected and preserved by adding 4% glutaraldyhyde in 0.1 M phosphate solution at pH 7.2 and dehydrated with ethanol for further SEM and EDX analyses (Erdei *et al.*, 2008).

3.6 EPS Analysis

In this study, EPS in mixing liquid of MBR tank was extracted by using sonication method, 15 mL of sludge was sonicated using sonicator (Ultrasonic processor, CV33) at 60 W for 2.5 min and then the sonicated liquid was centrifuged at 10000 g for 30 min at 4 °C. Finally, supernatant containing EPS was collected for measuring total protein and total carbohydrates content. The total content of protein in EPS was measured by the modified Lowry method (1995) using bovine serum albumin and humic acid as the respective standards. The total *carbohydrate* content of the *EPS* was determined using the phenol-sulfuric acid procedure (Dubois *et al.*, 1956*)*. Figure 3.7 shows the procedure of EPS analysis.

3.7 Membrane Cleaning

Membrane cleaning was required when TMP was increased up to 30 kPa. The procedure of membrane cleaning was commenced by disconnecting the suction lines from the membrane modules, and then the membranes were taken out from the reactor in order to remove the cake layer on membrane surface by shaking in a 5 L plastic cylinder which contains tap water. After that the membranes were immersed in a chemical cleaning tank for 6 hours. The cleaning solution was prepared by mixing 10% sodium hypochlorite (NaOCl) 50 mL with 5 L of distilled water. After 6 hours the membrane module was taken to remove cleaning solution by rinsing with tab water.

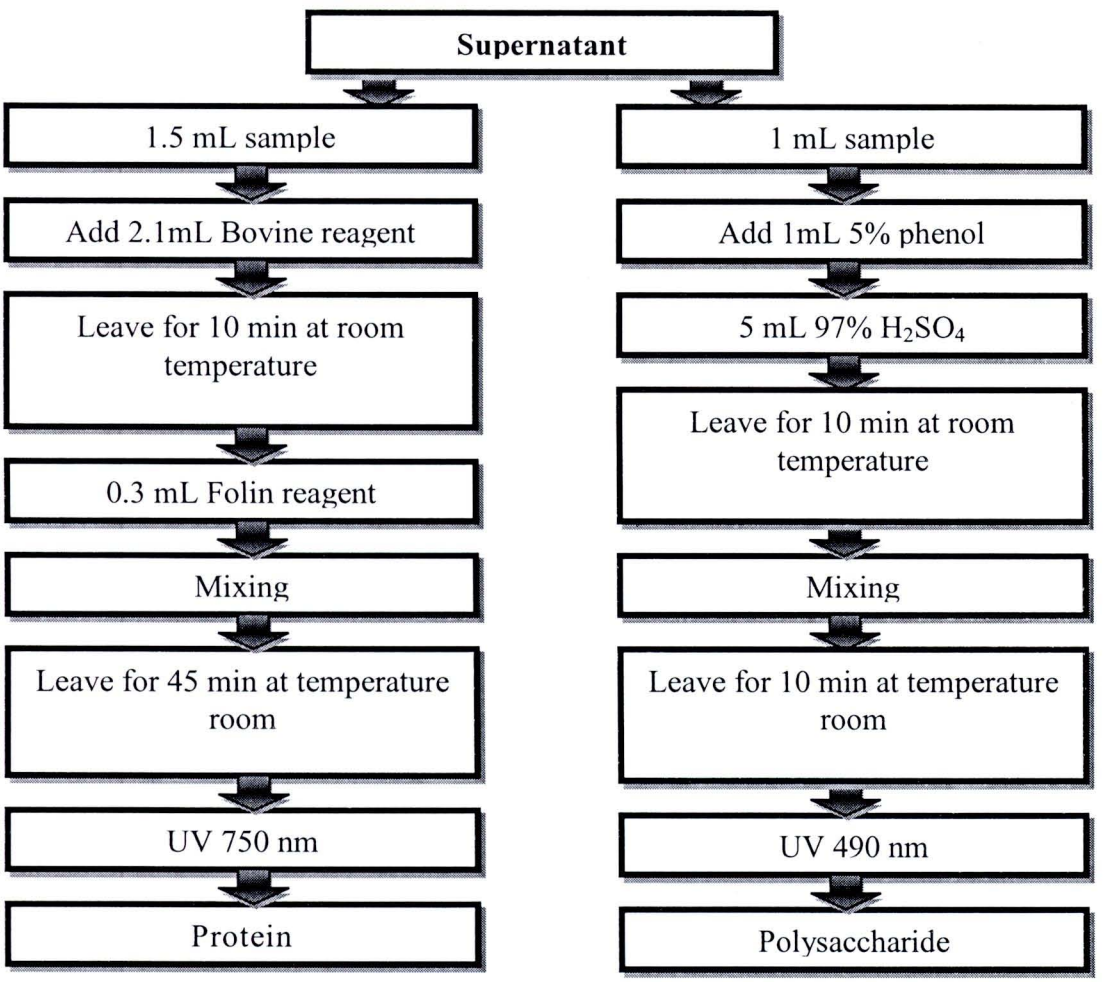

Figure 3.7 Procedure of EPS analysis.