Chapter 2

Experimentation

2.1 Chemicals

The VOCs used were toluene (99.5%) and methanol (99.8%) obtained from Merck, Germany.

2.2 Equipment and Instrument

2.2.1 Biofilter column

Three identical bench-scale biofilters were used to treat toluene, methanol, and mixtures of methanol and toluene from air streams. The biofilters were made of stainless steel and each consists of three equal segments connected in series. Each segment had a diameter of 5 cm and height of 30 cm. In order to support the filter bed and to ensure homogeneous radial distribution of the input gas, a stainless steel mesh was installed at the base of each section. These reinforced with stainless steel rods in order to bear the weight of the wet filter material. Two ports were placed in each segment, one for gas sampling and another one for media sampling. Bottom supported of column had a height of 8 cm for removing excess nutrients solution, and this part had inlet of contaminated air. The first and the second segments had two ports which were a medium sampling port and a gas sampling port, but the third section had three ports that they were a medium sampling port and two gas sampling ports for outlet of the second segment and outlet of the third segment.

The biofilter was fed by airflow provided by a continuous compressed air source. The major portion of the air was passed through a water column in order to become fully saturated. A secondary fraction of the main air was directed to a bubble unit containing the liquid VOC reagent (Figure 2.1). The flow rate of contaminated air was checked by Rota meter (102-05, Omega).

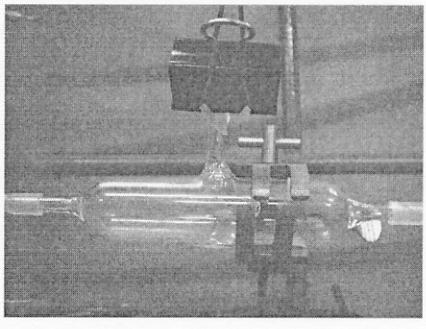


Figure 2.1 Glass tube for containing VOC.

2.2.2 *Filter material*

The biofilter media were a mixture of palm shells (0.5-1cm) and activated sludge (Kingfisher Holdings Ltd.) in proportion of 1:2 by volume. A pH buffer ($CaCO_3$) was added to the filter media when necessary. The media were kept for one night before packing in order to prevent the expansion of palm shells in the biofilter.

2.2.3 *Nutrient solution*

The nutrient solution was periodically distributed over the bed upper-surface to maintain an adequate level of bed filling relative humidity and to provide those nutrients necessary for the growth of microorganisms present in the biofilter. The composition of nutrient solution used is shown in Table 2.1.

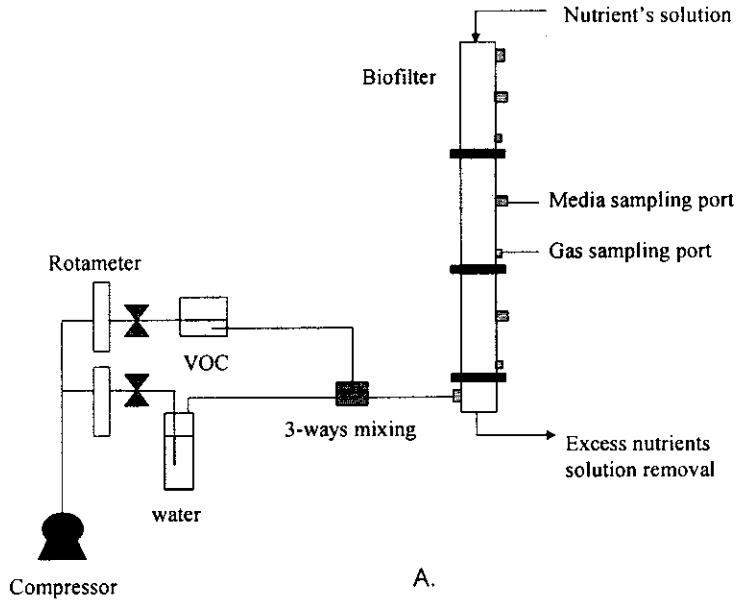
Table 2.1 Composition of one liter of the nutrient solution.

Compounds	Amount
KH_2PO_4	0.91 g
$Na_2HPO_4.12H_2O$	2.39 g
KNO_3	2.96 g
$(NH_4)_2SO_4$	1.97 g
$NaHCO_3$	1.5 g
$FeSO_4.7H_2O$	0.2 mg
$MgSO_4.7H_2O$	2 mg
$MnSO_4.H_2O$	0.88 mg
$Na_2MoO_4.2H_2O$	1 mg
$CaCl_2$	3 mg

2.3 Methodology

2.3.1 Preparation of system

The filter media was packed in stainless steel column at height of 20 cm in each segment. Then all three segments of column and bottom support were assembled as shown in Figure 2.2, and the compressed air was continuously emitted to column from bottom to top. Figures 2.3 to 2.5 present the three systems in this studied.



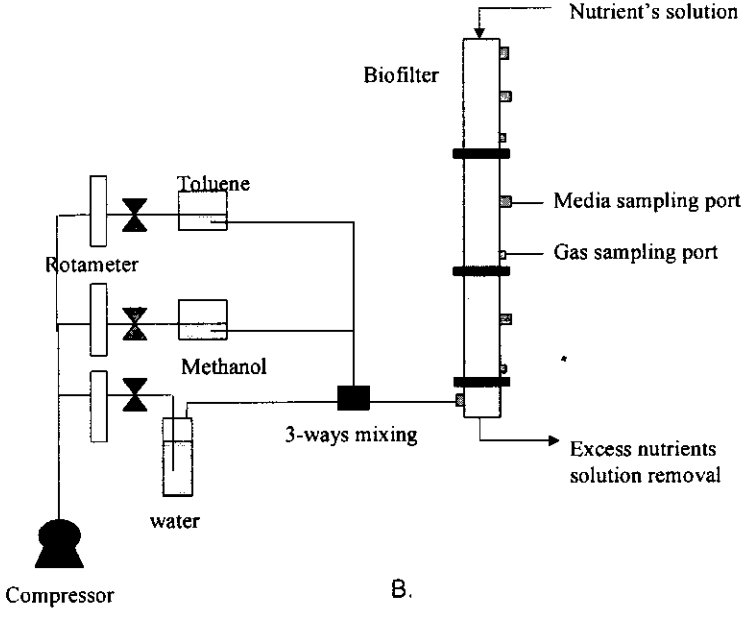


Figure 2.2 Experimental setup; A. pure VOC system and B. mixed VOC system.

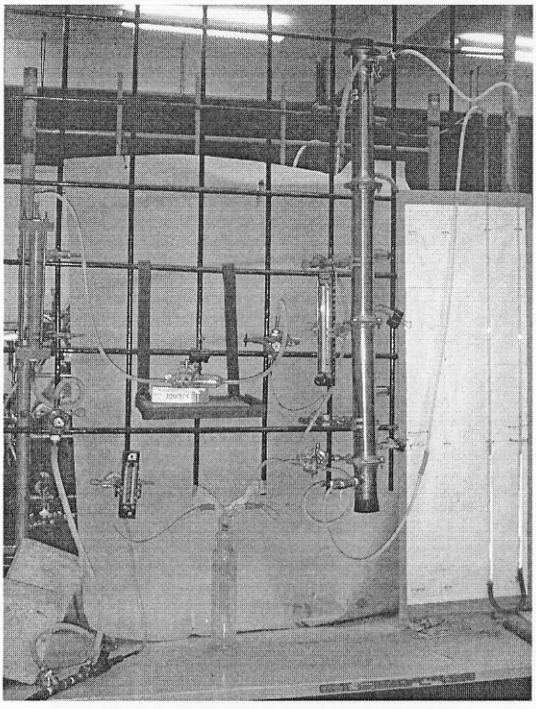


Figure 2.3 Toluene system.

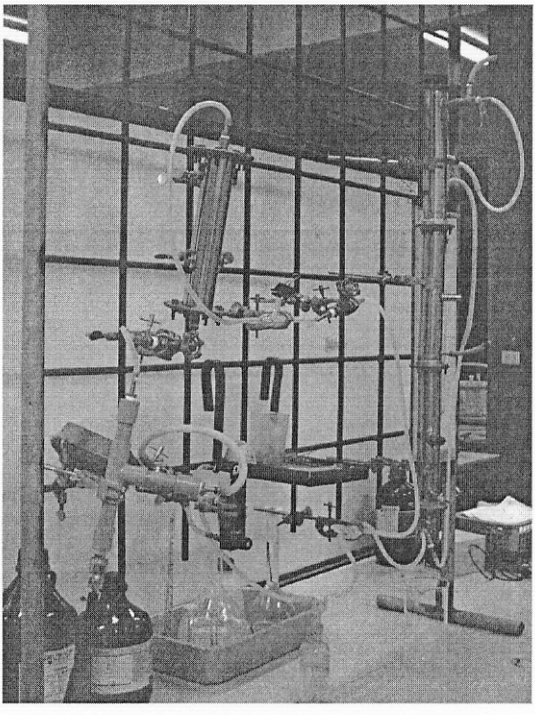


Figure 2.4 Methanol system.

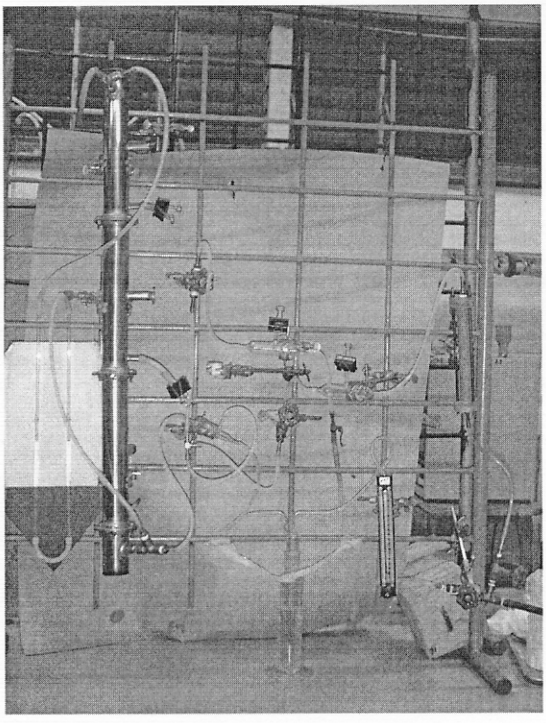


Figure 2.5 Mixture of methanol and toluene system.

2.3.2 Preparation of VOCs

Compressed air was separated into 2 lines and mixed at 3-ways mixing (Figure 2.6). The first line was passed through toluene and the second line was submerged in water for increasing the moisture. VOC was prepared at low concentration (0.3-2.5 g/m^3) that was checked by GC in each flow rate of 0.06, 0.12, 0.18, 0.24, and 0.45 m^3/h that was checked by Rotameter.

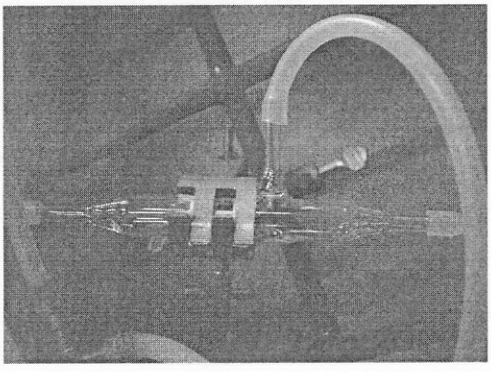


Figure 2.6 3-ways mixing.

2.3.3 Experiments

Each VOC prepared had been continuously emitted to column for 122 d at various flow rates and concentrations. Flow rates were studied at 0.06, 0.12, 0.18, 0.24, and 0.45 m^3/h. Concentrations of VOCs were studied in the range of 0.3-2.5 g/m^3. These conditions were changed when system was suitable. The sampling gas was taken at inlet and outlet ports by keeping in 100% polypropylene bag of 0.5 liter. Pressure drop between inlet and outlet, pH of three sections, relative humidity of three sections and temperature of three sections and ambient air were measured everyday in each system. At start up period, nutrient was supplied every day to stimulate the growth of microorganisms. After the systems went to steady state, nutrient was filled when relative humidity was less than 95%.

Nitrogen test was run to investigate that the system was dominated by biodegradation or adsorption by using nitrogen gas instead of air.

2.4 Analytical methods

2.4.1 Concentration of VOCs

Concentration of VOCs were analyzed by gas chromatography, GC (HP 6890, Hewlett Packard, F) equipped with a flame ionization detector and capillary column (HP1, crosslinked methyl siloxane) 30 meter of length, 0.25 μm of film thickness, and 0.32 mm of inlet diameter. Combustion gases were high purity hydrogen (flow rate 30 ml/min) and air zero grade (flow rate 400 ml/min). Make up gas was high purity nitrogen (flow rate 29 ml/min), and carrier gas was ultra high purity helium (flow rate 1 ml/min). Column temperature was increased from 30 °C to 70°C with a rate of 20 °C/min, then raised from 70 °C to 260 °C with a rate of 20 °C/min, and hold at 260 °C for 2 min. Split ratio was 100:1, and pressure between capillary column was 7 psi. Injector, oven and detector temperature were 180 °C, 70 °C, and 200 °C, respectively. The sampling gas was injected by gas tight syringe (Hewlett Packard). 4 μl of gas sampling was injected by gas tight syringe to GC. Peak area was compared with calibration curve for determining the concentration of VOC. Methanol and toluene were detected at 2.35 and 2.89 min, respectively. Concentration of methanol and toluene were determined using calibration curves.

2.4.2 Bed temperature, pH, Relative humidity, and Gas pressure drop

Bed temperature and relative humidity were monitored via AP-104 (Sila Research Co., Ltd., Thailand) at mid level while pH of the filter media was measured by indicator paper (Mersk, Germany). Gas pressure drop of the filter was measured by U-tube manometer.

2.4.3 Analytical Scan Electron Microscope

Samples for scanning electron microscopy (SEM) observation were fixed in 25% glutaraldehyde ($C_5H_8O_2$) in phosphate buffer for 1.5 h, washed twice with the same phosphate buffer, and post fixed in phosphate buffer containing 1% osmium tetraoxide (OsO_4) for 1.5 hours. Subsequently, the samples were washed twice with the distilled water prior to dehydration in a graded water-ethanol (50-100%) for 15 min in each step. The samples were finally dried in a critical point dryer and gold coated by a sputter coater. Examination was carried out in a digital scanning electron microscope (JSM-5800LV, JEOL).