CHAPTER III

EXPERIMENTS

3.1 Materials

Cetyltrimethyl ammonium bromide (CTAB), at a purity 99 %, was obtained from Rhone-Poulenc Company and was employed without further purification. Octadecyltrichlorosilane was received from Aldrich Chemical Company. Precipitated silica (Hi-Sil® 233) from PPG-Siam Silica had a reported BET surface area of 150 m^2/g. Toluene 99.5 %, sodium carbonate and trichloroethylene 99.5 % (TCE) were obtained from Carrlo Ebra Reagenti Company. Phenol (AR grade) and methanol (AR and HPLC grade) were received from Ajex Chemicals Company. Sodium hydroxide from Baker Company was used for adjusting the pH. Ozone was produced using an Ozone Generator OG20 of Verfahrenstechnik Universal GmbH Company. All solutions were prepared with distilled water.

3.2 Methods

3.2.1 Adsorption Measurements

The experimental procedure to measure the CTAB adsorption is summarized as follows:

1. Prepare an aqueous CTAB solution of 500 ml at neutral pH.
2. Mix 14 ml of solution with 0.7 g of silica particles in a 15 cm-long glass test tube.

3. Occasionally agitate the 30 °C mixture over a period of at least 4 days to allow the equilibrium adsorption of CTAB.
4. Measure the aqueous phase CTAB concentration (solutions were filtered) by using a Hewlett Packard Series 1050 high performance liquid chromatography (HPLC) with an Alltech column universal cation 7U 100 mm * 4.6 mm and an Alltech 350 conductivity detector.
5. Calculate the aqueous phase concentration by interpolating the CTAB calibration curve which is obtained from the CTAB peak areas.
6. Determine the amount of CTAB adsorbed on the silica by a mass balance and then calculate the adsorbed CTAB per gram of silica (μmole/g).

3.2.2 Chemically Bonded Monolayer of Hydrocarbon on Silica

The basic procedure to construct the chemical bonding of octadecyl-trichlorosilane (ODS) (Ogawa et al., 1994) is given as below:

1. Mix ODS, 6 g of silica with 180 ml of toluene in 500 ml round bottom flask equipped with condenser.
2. Reflux the mixture for 12 hr, then add 24.85 ml of methanol and reflux again for 3 hr.
3. Filter the mixture through a membrane filter and wash the solid with 400 ml of distilled water follow by 400 ml of toluene.
4. Wash the solid with toluene by mean of a soxhlet extractor for a day and then dry it over night at 80 °C.

5. Measure the spectra of the silica with toluene by mean of the silica treated ODS on a Bio-Rad FTS-45A infrared spectrometer by the absorbance technique as a KBr disc.
6. Measure TG-DTA of ODS silica from room temperature to 850 °C with a Netzsch STA 409 EP (30 mg of sample, alumina crucible and dynamic atmosphere of air).
7. Determine the amount of ODS bonded to the silica by weight loss from TG curve and also calculate the bonded ODS per gram of silica (μmole/g).

3.2.3 Adsolubilization Measurements

The adsolubilization of phenol and trichloroethylene (TCE) into CTAB admicelles and ODS monolayer was determined by an HPLC. The experiment procedure for phenol is given as follows:

1. Prepare 10^5 μM of phenol solution in a 1000 ml volumetric flask.
2. Inject 300 μl of the phenol solution into a 15 cm-long glass test tube containing 14 ml of CTAB solution and 0.7 g of silica which has equilibrated for a period of at least 4 days.
3. Occasionally agitate the 30 °C mixture for 2 hr, then withdraw samples from the solution by syringe filtration.
4. Measure the phenol concentration in aqueous phase by using a Hewlett Packard Series 1050 HPLC with Hewlett Packard column spherisorb ODS2 5 μm, 125 mm * 4 mm and a U.V. detector 1050 Series.
5. Calculate the concentration of phenol in admicelles by subtracting that in the aqueous phase from the total concentration.

The measurements of adsolubilized phenol in monolayer were carried out in a similar processes. The 0.7 g of ODS silica is used instead of pure silica.

TCE adsolubilization measurements were continued in the same fashion with phenol. The analytical reagent grade 99.5 % of TCE was employed in this experiment by injecting 10 μl of TCE into the 14 ml of solution and 0.7 g of silica and/or 0.7 g of ODS silica.

3.2.4 Stability Measurements

The effect of agitation on desorption of admicelles of CTAB on silica is shown as below:

1. Mix 0.1 g of silica that has the adsorbed CTAB 300 μmoles/g silica with 50 ml of distilled water in the 250 ml beaker.
2. Place it in the temperature control bath (DT Hetotherm) and agitate with mechanical stirrer (Janke & Kunkel IKA®-Labortecnik) with speed No.1 for 1/2 hr at 25 °C.
3. Measure the desorbed CTAB concentration in solution by HPLC with conductivity detector and find the % desorption of CTAB on silica.
4. Repeat step 1-3 but varying agitation speed.

The effect of pH was done in the same steps with effect of agitation speed but the pH value was varied instead of the speed. The effect of temperature and agitation time were carried out in a similar method.

For the ODS silica, it was weighed 0.1 g and continued in the same fashion with admicelles on silica. The TG-DTA analyzers (Netzsch STA 409 EP) was used for measuring the weight loss of ODS group and determine the % desorption of bonded ODS on silica.

The stability of ODS silica under ozone condition was studied and the experimental procedure is given as below:

1. Mix 0.15 g of ODS silica with 100 ml of distilled water in a 500 ml wide neck reaction flask.
2. Ozone was generated from ozone generator OG20, Verfahren-stechnik Universal GmbH, which was analyzed by ozone analyzer OM40 and fed into the solution with flow rate 15 l/hr together with stirring the solution at 500 rpm and 25 °C (±0.5)(see Figure 3.1).
3. Filter the solid and dry it over night at 80 °C.
4. Determine the % carbon on the solid by elementary analyzer, Perkin-Elmer, and find % oxidation of ODS by ozone.
5. Repeat step 1-4 and vary the concentrations of ozone and pH values of the solution.

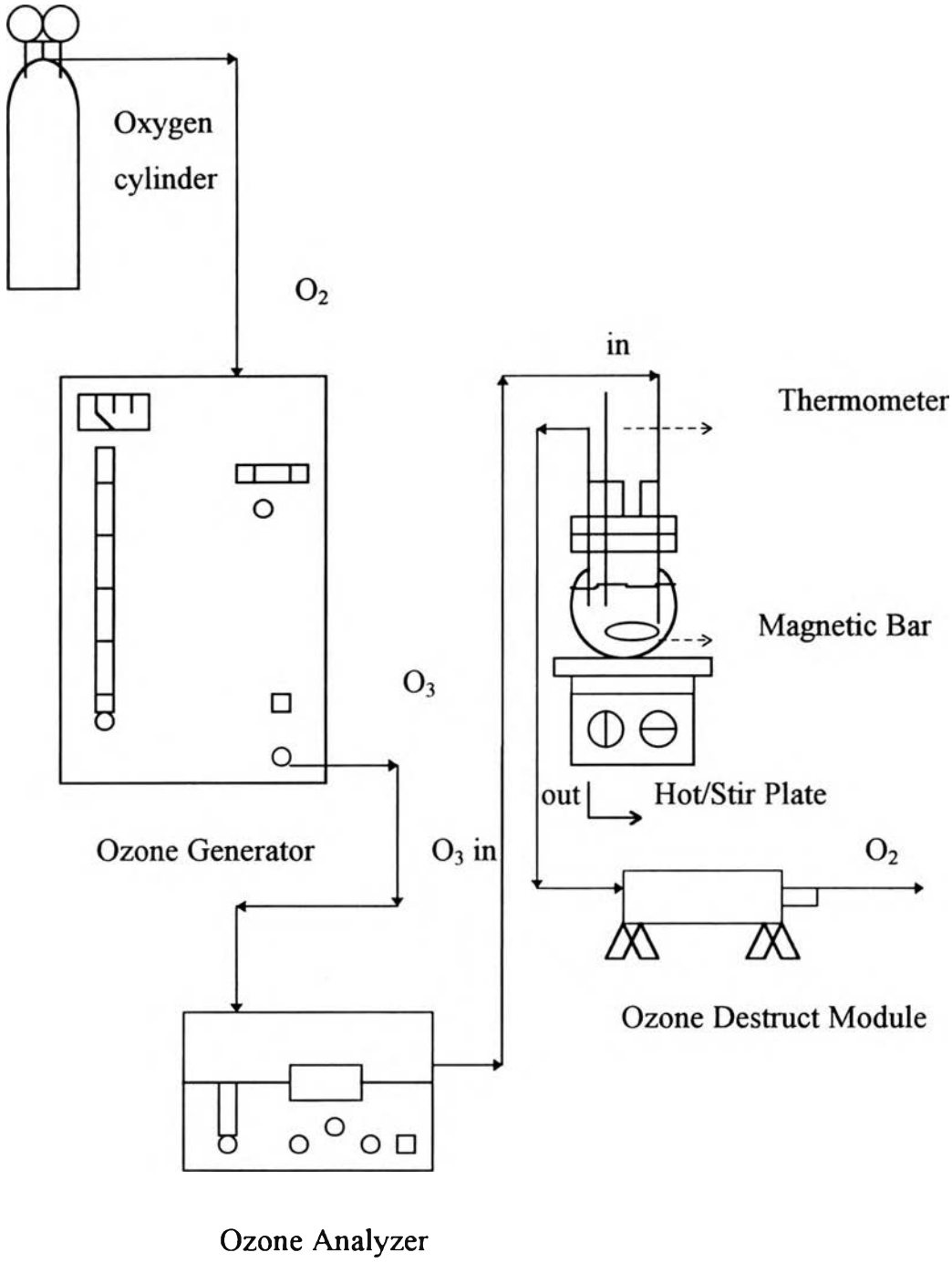

Figure 3.1 Experiment apparatus in ozone oxidation.