

## CHPATER III

### EXPERIMENTAL

#### Chemicals, Equipment and Glassware

##### Chemicals

1. Silica, Silicon dioxide, SiO<sub>2</sub> (99.99% pure), fumed, 0.007 micron, purchased from Aldrich Chemical Company, Inc., was used as received.
2. Silica, SiO<sub>2</sub> (98.50% pure), HI-SIL 928, 10.97 micron, provided by PPG-Siam Silica Co., Ltd., was used as received.
3. Rice husk ash, burnt at 800°C, silica 96.74%, provided from Maboon Klong Rice mill, 13.47 micron by milling Current JET Crusher CJ-10, was used as received.  
Ethylene Glycol (EG), commercial grade from Carlo Erba Reagent Co., Ltd. were purified by azeotropic distillation.
4. Triethanolamine (TEA) and Diethanolamine (DEA), commercial grade, provided by Union Carbide (Thailand) , were purified by azeotropic distillation.
5. Other chemicals of analytical grade from Baker Analytical Co., such as Diethylether, Acetonitrile, Methanol, were used as received.

### Equipment and Glassware

3 necked round bottom flask, magnetic stirrer, magnetic bar, heating mantle, boiling chips, soxlet, condenser, oven, filtering system, analytical balance and other general laboratory glassware and equipment.

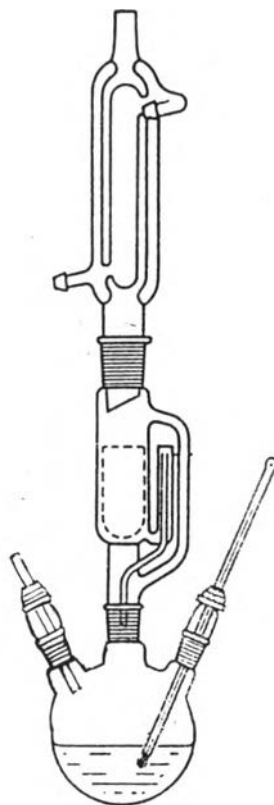
### **Instruments**

1. Fourier Transform Infrared Spectrometer (FT-IR), Model Impact 410
2. Fourier Transform Nuclear Magnetic Resonance Spectrometer, Model AC F200 (200 MHz), Bruker Spectrospin ( $^1\text{H-NMR}$ ,  $^{13}\text{C-NMR}$ )
3.  $^{29}\text{Si}$  Nuclear Magnetic Resonance Spectrometer, JEOL (500 MHz)
4. Thermogravimetric Analyser, TAC7, Perkin Elmer
5. Elemental Analyser (EA), Series II CHNS/O Analyzer 2400, Perkin Elmer
6. X-ray Fluorescence Spectrometer, OXFORD ED2000
7. X-ray Diffraction Spectrometer, JEOL Model JDX-8030, Japan

### **Experimental Procedure**

#### 1. The Synthesis of Polysiloxane

Synthesis was carried out in a 250 ml three-necked round bottom reaction flask equipped with soxlet, a thermometer, a nitrogen-gas inlet (Figure 3.1) and heated by heating mantle.



**Figure 3.1** Reactor

### 1.1 Synthesis of products using triethanolamine (TEA)

100 mmol of silica (6.00 g of  $\text{SiO}_2$  from (1), 6.09 g of  $\text{SiO}_2$  from (2) or 6.22 g of rice husk ash), TEA (which was varied from 25, 50, 100 to 150 mmol; 3.725, 7.45, 17.90 and 18.625 g, respectively), and 50 ml of EG were placed in a 250 ml three-necked reaction flask, boiling chip was added and the reaction was set up as in figure 3.1. The flask was placed in a heating mental at various temperature from 100-200°C and reaction times of 12-54 hour. The reaction mixture was allowed to cool to room temperature and filtered to remove unreacted  $\text{SiO}_2$  or rice husk ash which was washed with methanol, dried in oven at 100°C for 10 hour, and weighed to calculate the percent conversion of silica.

EG was removed from the yellow filtrate under reduce pressure, to give a yellow viscous solution. Product was purified by precipitating with diethylether:acetonitrile (50:50). The powder was isolated by filtration, dried in a vacuum descicator and then characterized by FT-IR,  $^1\text{H-NMR}$ ,  $^{13}\text{C-NMR}$ ,  $^{29}\text{Si-NMR}$  spectroscopy, Elemental Analysis and Thermal Analysis.

### 1.2 Synthesis of products using diethanolamine (DEA)

The same procedure as described in the process above was carried out, except that the reaction used DEA. The amount was varied from 25, 50, 100 and 150 mmol; 2.625, 5.25, 10.50 and 13.125 g, respectively, reaction temperature was varied from 100-200°C, reaction time 12-54 hour.

## 2. Characterization

Products were subjected to various instrumental analyses as shown below

### 2.1 Study of the Functional Groups

Preparation of solid powder products were prepared by the pressed disc technique in a form of pellet KBr, and scanned with a to determine the functional groups of the compounds by FT-IR spectrometer.

### 2.2 Study of the Structure

Solid powder products were dissolved in deuterated dimethyl sulfoxide ( $\text{d}_6\text{-DMSO}$ ) and analysed by  $^1\text{H-NMR}$ ,  $^{13}\text{C-NMR}$ ,  $^{29}\text{Si-NMR}$  Spectrometer, Elemental Analyser and X-ray Fluorescence Spectrometer. Some

solid powder products were not soluble so they were characterized by Solid State NMR Spectrometer, Elemental Analyser and X-ray Fluorescence Spectrometer.

### 2.3 Study of Thermal Properties of the Products

Decomposition Temperature (Td) was determined by Thermogravimetric Analyser. 5-20 mg of the sample was placed into a platinum pan and heated in N<sub>2</sub> and was equilibrated at 50°C, scanning with a heating rate of 20°C/min until 900°C