

# CHAPTER III

## EXPERIMENTAL



### 3.1 Chemicals

1. Activated alumina (base), $\text{Al}_2\text{O}_3$	MERCK
2. Activated alumina (neutral), $\text{Al}_2\text{O}_3$	MERCK
3. Anhydrous ferric chloride, $\text{FeCl}_3$	CARLO ERBA
4. Methanol, $\text{CH}_3\text{OH}^*$	ITALMAR
5. Nitrogen, $\text{N}_2$	TIG
6. Potassium persulfate (KPS), $\text{K}_2\text{S}_2\text{O}_8$	MERCK
7. Polyethylene (PE)*	TPI
8. Polypropylene (PP)*	TPI
9. Poly(vinyl chloride) (PVC)*	TPC
10. Poly(vinyl pyrrolidone): K40 (PVP)	SIGMA
11. Sodium chloride, $\text{NaCl}$	MERCK
12. Anhydrous Sodium sulfate ( $\text{Na}_2\text{SO}_4$ )	FLUKA
13. Styrene*	Eternal Resin

\* The commercial grade chemicals

### 3.2 Glasswares and Equipments

1. Beaker
2. Büchner funnel
3. Condenser
4. Glass stopper
5. Gas inlet, outlet tube
6. Magnetic stirrer hotplate
7. Mechanical stirrer
8. Multimeter
9. Three-necked, round bottom flask
10. Water bath
11. Water pump
12. Temperature controller bath

### 3.3 Instruments

1. Fourier Transform Infrared Spectrometer (FT-IR)
  - (1) NICOLET Impact 410
  - (2) Perkin Elmer 1760x
2. Scanning Electron Microscope (SEM)  
JEOL, JSM-6400



### 3.4 Purification and preparation of materials

#### 3.4.1 Styrene monomer

Styrene was firstly washed twice with 10 % NaOH solution and then with distilled water. It was further purified further by eluting through the column packed with activated alumina ( $\text{Al}_2\text{O}_3$ ) base and neutral. Purified styrene was kept in the bottle containing anhydrous  $\text{Na}_2\text{SO}_4$  and stored at 5 °C for no more than 2 weeks before use.

#### 3.4.2 Pyrrole monomer

The pyrrole monomer was distilled at atmospheric pressure as colorless liquid. The distillate was collected in the glass bottle and then sealed instantly. The sealed pyrrole bottle was stored at - 4 °C for no more than 1 week before use.

#### 3.4.3 Methanol

Commercial grade methanol was purified by distillation before use.

#### 3.4.4 Oxidant solution

To obtain 2.5 M oxidant solution of  $\text{FeCl}_3$ , 20.2775 g of anhydrous ferric chloride was dissolved in 50 ml methanol at 0 °C. The precipitates and impurities were filtered of and the clear orange solution was obtained. The oxidant solution was kept at -4 °C and used within 24 hours.

### 3.5 Synthesis of micro-particles polystyrene [44]

The styrene monomer (30 ml), potassium persulfate (0.30 g), sodium chloride (0.35 g) and water (150 ml) were added into the three-necked flask, equipped with a magnetic stirrer, gas inlet/outlet tubes for flowing nitrogen gas. The reaction was allowed to proceed for 10 hours at 70 °C using hot water bath. The slurry mixture was poured into a 100 ml of 1:1 mixture of water and methanol. The mixture was cooled at room temperature overnight. Then, it was centrifuged and the supernatant was discarded. The fine precipitate, like cream, was redispersed in methanol and dried at room temperature in desiccator.

### 3.6 Synthesis of polypyrrole coating composite

The host polymer (2 g) (PS, PVC, PE, or PP) and 50 ml 2.5 M FeCl<sub>3</sub> solution was added into 100 ml three-necked flask, equipped with a drying tube and gas inlet tube for bubbling nitrogen. The mixture was magnetically stirred while the temperature was kept at 0 °C by using ice and salt bath (Figure 3.1). The reaction was allowed to proceed for 30 minutes before the pyrrole monomer was added and then stirred for another 30 minutes. At the end of reaction, a 100-ml methanol was added to quench the reaction. The polymer composite was filtered and washed with methanol until the filtrate was colorless. The polypyrrole composite was then dried in a desiccator at room temperature.

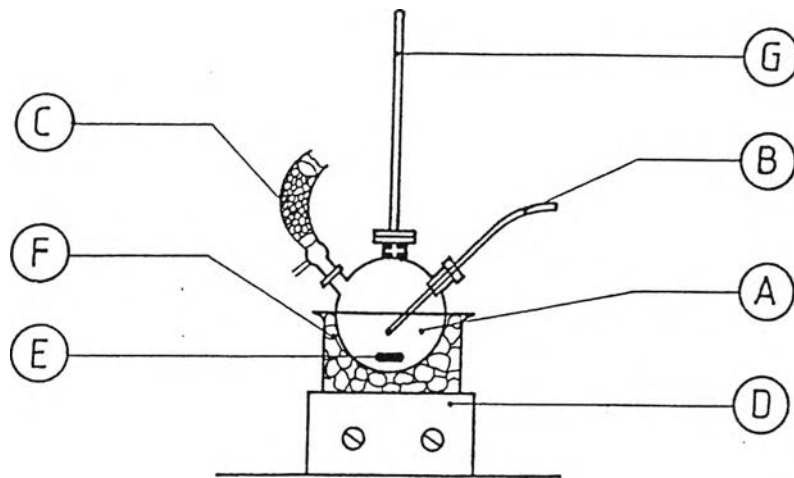


Figure 3.1 Polymerization of polypyrrole composite set up

- |                      |                     |
|----------------------|---------------------|
| (A) Three-neck flask | (E) Magnetic bar    |
| (B) Gas inlet tube   | (F) Ice bath        |
| (C) Drying tube      | (G) Stand and clamp |
| (D) Magnetic stirrer |                     |

### 3.7 Synthesis of dispersion polypyrrole

Poly(vinyl pyrrolidone) (5 g) was added into 50 ml three-neck flask containing 2.5 M  $\text{FeCl}_3$  solution. This procedure was similar to the preparation of polypyrrole coating composite (Section 3.6).

### 3.8 Determination of condition effects

#### 3.8.1 Polypyrrole coating composites

##### 3.8.1.1 pyrrole volume

The polymerization of this section was carried out under the reaction conditions similar to those in Section 3.6. The pyrrole monomer was changed by the weight ratio of pyrrole: host polymer of 0.1, 0.2, 0.5, 0.8 and 1 to study the effect of pyrrole on the composite polymerization.

##### 3.8.1.2 host polymer type

In the same manner, the polymerization was carried out under the reaction similar to those in Section 3.6. Various types of host polymer were used to study the effect of each host polymer.

##### 3.8.1.3 reaction time

The polymerization was carried out under the reaction conditions similar to Section 3.6. The reaction time was varied from 1 to 3 hours to study the effect of reaction time on the occurrence of polymer composites.

##### 3.8.1.4 solvent

To investigate the effect of the reaction media between aqueous and nonaqueous, the oxidant solution was prepared in aqueous solution

and methanol solution, respectively. Anhydrous ferric chloride was dissolved in distilled water to obtain 2.5 M concentration and then use to polymerize polymer composite in the same as Section 3.6.

#### 3.8.1.5 temperature

The polymerization was carried out in the reaction condition similar to Section 3.6 but the temperature was changed from -20 to 0°C. The 0.5 pyrrole: host polymer weight ratio was used as a constant. Polystyrene powder is the selected host polymer for this section. The temperature of the reaction was controlled by various techniques due to the high efficiency water bath that could control the temperature to the minus value level, was not available. The 0 °C temperature was fixed by using ice and salt bath and at below than 0 °C, dry ice in methanol was used.

### 3.8.2 Dispersion polypyrrole

#### 3.8.2.1 pyrrole volume

Similar to the reaction condition in Section 3.7, the polymerization was carried out in the presence of PVP. Except the pyrrole amount that was changed from 0.1 to 0.8 weight ratio to study the effect of pyrrole monomer on the composite polymerization.

### 3.8.2.2 reaction time

The polymerization was carried out in the same manner as in Section 3.7. The other parameters were kept constant except the reaction time. The reaction time was changed from 1 to 3 hours. These polymer products were polymerized in water.

### 3.8.2.3 solvent

Similar to Section 3.7, the polymerization was accomplished with various solvents. Those of solvent were methanol, water, and 50% methanol/water, which were used to synthesize the polypyrrole samples.

## 3.9 Characterization

### 3.9.1 Electrical conductivity measurement

The polypyrrole composites were transformed into a thin disc by pressing under three tons of hydraulic force in evacuable die for 5 minutes. Three samples of each composite are prepared for conductivity measurement.

The electrical conductivity of each sample was measured by van der Pauw method, which was clearly described in Appendix A. Three samples were measured under the same contact points and the average value of these measurements was taken.



### 3.9.2 Conductive stability of polymer composite disc

Some of polypyrrole composite samples were subjected to the conductivity measurement at several intervals of time using van der Pauw method, and then they were next measured several times for their conductivities every 2 weeks until the measurement reached 10 weeks. These measurements show the stability of electrical conductivity in each sample.

### 3.9.3 Fourier transform infrared spectroscopy

The absorption spectra of polypyrrole samples were obtained by using Attenuated Total Reflectance Fourier Transform Infrared (ATR FT-IR) technique. The polypyrrole composites must be prepared to the latex form and then cast on the ATR prism before analyzing. The ATR FT-IR method to use to characterize the polymer composite was described in Appendix C.

### 3.9.4 Scanning electron microscopy

Morphology of polymer particles was investigated by Scanning Electron Microscope technique. Polypyrrole samples were prepared by firstly coating with gold in order to discharge the electron from electron beam in electron microscope. The photographs of polymer samples were then taken.