CHAPTER III
EXPERIMENTAL METHODS

3.1 Chemicals:

3.1.1 Monomers.

C_4H_5N, pyrrole monomer, from Fluka, was purified by distillation under reduced pressure and kept at -10°C before use.

Mw = 67.09, b.p. = 129-131°C, d^{20} = 0.966 g/cm^3

3.1.2 Oxidizing agent.

$FeCl_3$, ferric chloride, from Carlo Erba.

Mw = 162.22

3.1.3 Matrix polymer.

Poly(vinyl alcohol) (PVA), from Baker Analyzed.

Mw = 11,000-31,000

3.1.4 Plastic films substrate.

Poly(vinyl chloride) (PVC), from Thai Plastic Chemical (public) Co., Ltd. (TPC)

Mw = 45,000, d^{20} = 1.511 g/cm^3

Polypropylene (PP), from Thai Petrochemical Industry (public) Co., Ltd. (TPI)

MI = 11.4 g/10min (Mw = 44,000), d^{20} = 0.908 g/cm^3

Low Density Polyethylene (LDPE), from TPI

MI = 5.90 g/10min (Mw = 45,000), d^{20} = 0.922 g/cm^3

3.1.5 Other chemicals.

Distilled water

Iodine crystal from Fluka, kept in dark

3.2 Equipments and Glasswares.

Optical Microscope; Olympus reflected microscope OLYMPUS BX60M.

Elemental Analysis (EA); Perkin Elmer PE 2400 Series ll: option CHN

Fourier-Transform Infrared Spectroscopy (FTIR); Nicolet Impact 410

Differential Scanning Calorimeter (DSC); NETZSCH DSC 200,

Thermogravity Analysis (TGA); NETZSCH TGA 200.

UV-visible spectrophotometer; Hitachi U-2000

Vacuum Oven; Gallenkamp.

Refrigerator; Sanyo C-B series.

Desiccator ∅ 8 inches.

Oven; Memmert.

Tensile Strength and Elongation; Universal Testing material Machine;

Lloyd 500

Vacuum pump; Eyela A-3S

Mechanical Stirrer; Heidolph MR 3001

Other general laboratory glasswares and equipments.

3.3 Procedures.

3.3.1 Preparation of conducting plastic films by CVD of pyrrole.

The conducting plastic film was prepared by CVD of pyrrole. The equipment was set-up as shown in Figure 3.1. The conducting plastic film can be also prepared by various plastic films such as PVC PP and LDPE. The variable parameters in the preparation of conducting plastic films were shown in Table 3.1.

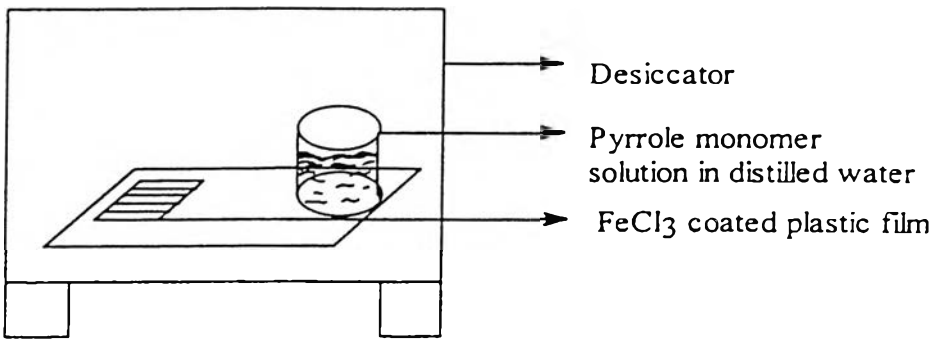

Figure 3.1 Apparatus for preparation of conducting plastic film by CVD of pyrrole.

Table 3.1 Preparation of conducting plastic film by CVD of pyrrole with various parameters.

Parameters	Variable values
$FeCl_3$ concentration (% in H_2O)	0, 5, 10, 15, 20, 25
C_4H_5N monomer concentration (% in H_2O)	0, 5, 10, 15, 20, 25
Reaction temperature (°C)	-15, 0, 10, 30, 50, 70
Reaction time (hours)	0, 5, 10, 15, 20, 25
Polymer matrix PVA (g)	1
Reaction condition	Vacuum
Plastic film substrate, thickness 40 μm	PVC, PP, LDPE

Polypyrrole was coated onto plastic films in vacuum desiccator by CVD as shown in Figure 3.1. The procedure for preparation of conducting plastic film by CVD of pyrrole was as follows:

1 g of PVA was dissolved in 10 mL of distilled water, then the solution was stirred and heated at 40-50°C until the clear solution (I) was obtained. 5 g of $FeCl_3$ (5%w/v) were dissolved in 10 mL of distilled water and stirred with mechanical

stirrer for 3-4 hours, then the solution was filtered for 2-3 times and allowed to settle in the dark for 24 hours after that the solution was filtered for 3 times again until to give the clear solution (II). The solution (I) and (II) were mixed together with stirring until the homogeneous solution was obtained. Five drops of the resulting solution was coated onto the surface of various plastic films which was supported on a slide, 2 cm x 8.5 cm, as show in Figure 3.1 then the prepared plastic films were dried in vacuum oven at 50°C for 3 hours. The prepared plastic films were placed in desiccator containing pyrrole monomer solution and the desiccator was reduced pressure using vacuum pump for 2 minutes. The desiccator was kept in various conditions as shown in Table 3.1. Finally, the resulting conducting plastic films were removed from the desiccator and dried by vacuum pump about 2 minutes for quenching reaction and subjecting the resulting conducting plastic films immediately to measure the electrical conductivity.

3.3.2 Doping conducting plastic film with iodine vapor.

The procedure for preparation of conducting plastic film by CVD of pyrrole and iodine vapor doping were shown in Figure 3.2 utilizing equipment setup as shown in Figure 3.3

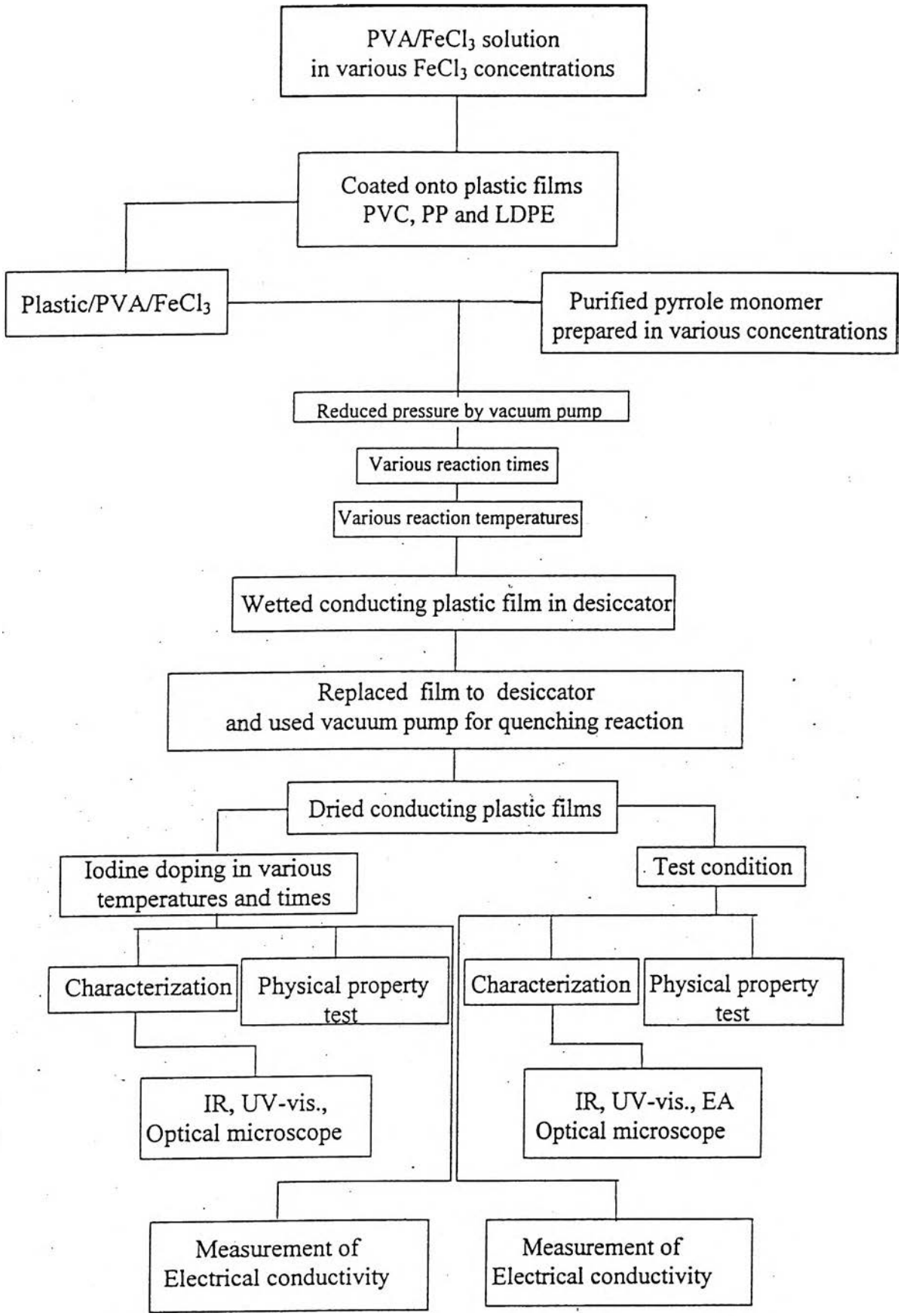

Figure 3.2 Diagram for preparation of conducting plastic film by CVD of pyrrole and characterization.

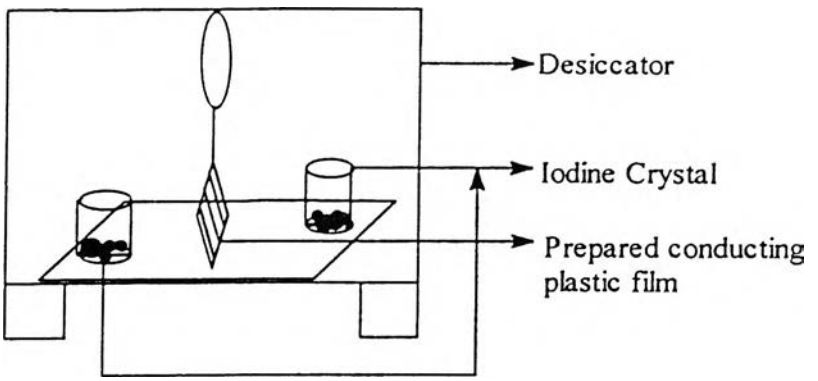

Figure 3.3 Apparatus for iodine vapor doping on prepared conducting plastic films.

The PP/PPY conducting plastic films and two 100 mL beakers each containing 3 g of iodine crystal were placed in desiccator as shown in Figure 3.3 then the desiccator was reduced pressure and kept at 30°C for 30, 60, 90, and 120 minutes and kept to 60 minutes for iodine doping at the temperature of 0, -15 and 70°C. Then, the prepared conducting plastic films were characterized to determine the physical properties and electrical conductivity.

3.3.3 Determination of electrical conductivity of conducting plastic films [see Appendix A].

The electrical conductivity of prepared conducting plastic film by CVD of pyrrole was measured immediately by van der Pauw method after preparation at room temperature.