CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Polymer Chemical

- 2,3-butanedione
- Hydrazine sulfate ($H_2NNH_2.H_2SO_4$)
- Sodium carbonate (Na_2CO_3) powder
- Ether (diethyl ether)
- Iodine (I_2) anhydrous, beads
- Ethylene Propylene Diene Rubber (EPDM)

3.1.2 Solvent

- n-butanol AR
- Chloroform ($CHCl_3$) AR
- Hexane

3.2 Equipment

- Fourier transform infrared spectrophotometer (FTIR)
- Scanning Electron Microscope (SEM)
- Rheometer to determine G′(ω) and G″(ω)
- Electrical conductivity meter
- Labolatory test sieve (APERTURE 56 μm)

3.3 Methodology

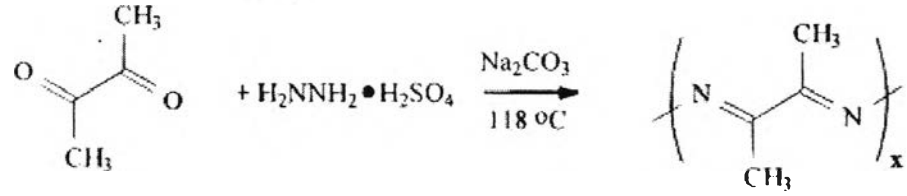
3.3.1 Synthesis of Conductive Polymer, Permethylpolyazine (PAZ)

3.3.1.1 Polymer Synthesis

Typically, 5.0 g of hydrazine sulfate, $H_2NNH_2.H_2SO_4$ (0.038 mol) was added to 5 mL of H_2O. Then 4.07 g of sodium carbonate, Na_2CO_3 (0.038

mol) was added, and the mixture was stirred at room temperature until the CO_2 formation ceased. Then 40 mL of n-butanol was added to the flask (Euler W.B., 1996).

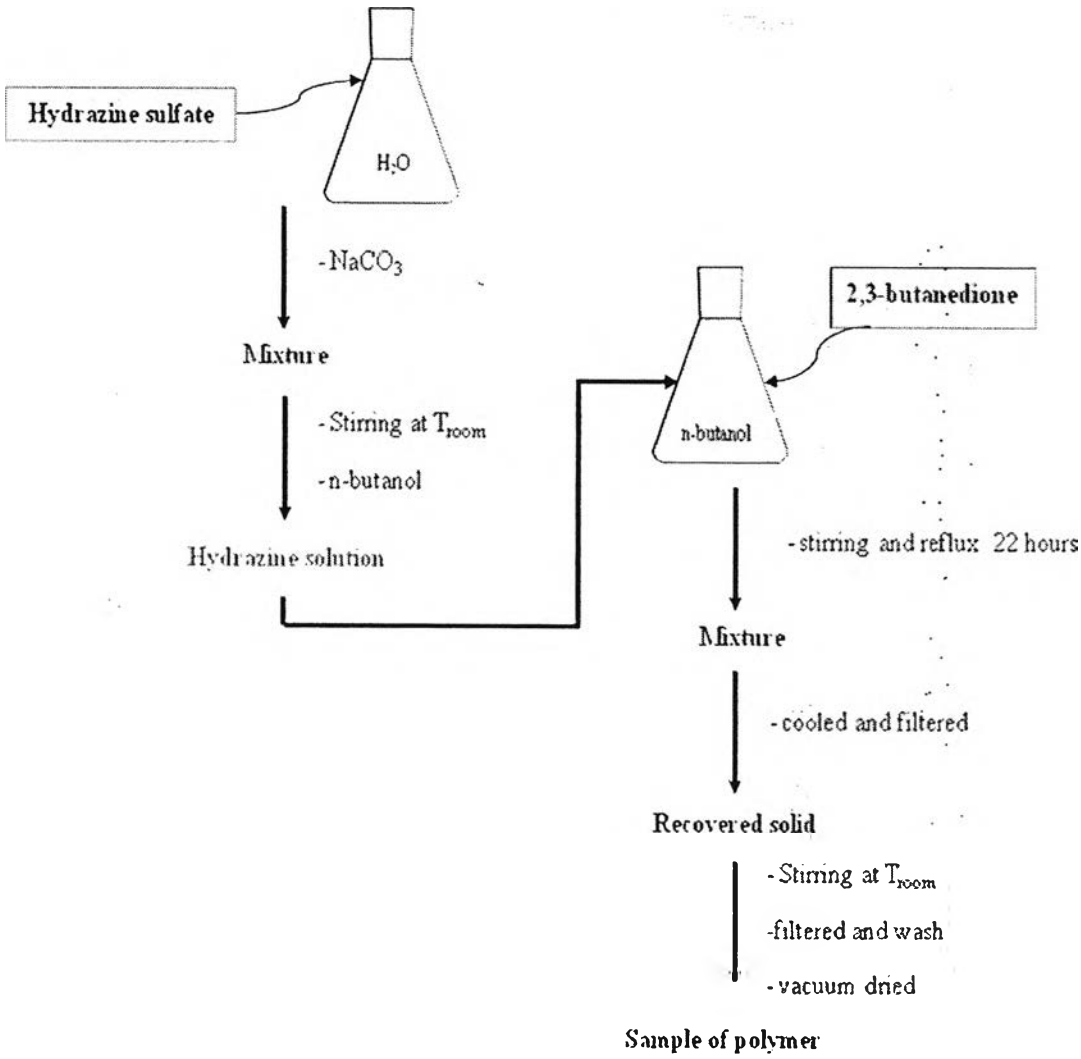
A separate solution of 3.4 mL (0.038 mL) of freshly distilled 2, 3-butanedione in 20 mL of n-butanol was prepared and added to the hydrazine solution slowly over the course of about 5 min. The entire mixture was then stirred and brought to reflux for 22 h. Then it was cooled on ice and filtered. The recovered solid was stirred in room temperature water for about 3 h to remove Na_2SO_4, filtered, washed with ether, and vacuum dried; this yields 3.1 g (100%) (Euler W.B., 1996).



2,3-butanedione

Permeyhylpolyazine (PAZ)

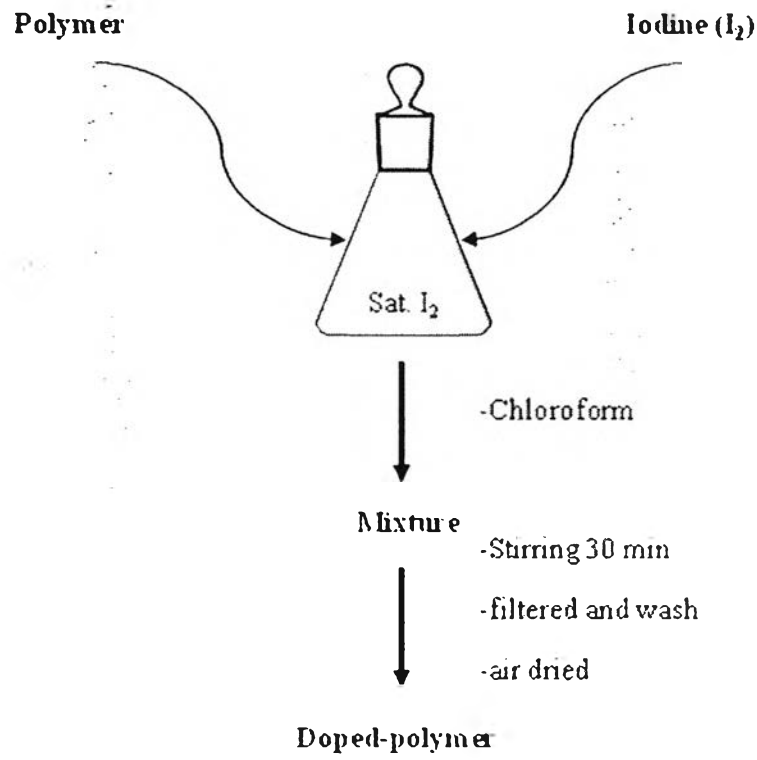
Flow chart of preparation of polyazine



3.3.1.2 Doping Permethylpolyazine

Normally, 0.30 g of polymer (3.7 mmol repeat unit) was added to a 125 mL Erlenmeyer flask, and a weighted amount of I_2 was added to the flask. It was then corked, and the iodine was allowed to saturate in the chamber for about 1 hr. Then 10 mL of chloroform, $CHCl_3$, was added, the flask recorked, and the mixture was stirred for 24 hr. Then it was filtered, washed with $CHCl_3$, and air-dried (Euler W.B., 1996). The doped-permethylpolyazine (D_PAZ) powder was sieved with a mesh particle size of 56 μm before used.

Flow chart of doping polyazine



3.3.2 Preparation of the PAZ/EPDM Blend

3.3.1.3 Blending of permethylpolyazine with ethylene propylene diene rubber

The blend was prepared by mechanical blending (solution casting blend) of sieved highly doped permethylpolyazine with a ethylene propylene diene rubber. We dissolved 3.5 cm^3 of ethylene propylene diene rubber in 35 cm^3 of hexane and stirred for 24 hours. Then we added highly D_PAZ powder and stirred the mixtured for 24 hours. Concentration of D_PAZ in EPDM prepared were 5, 10, 15, and 20vol.%. We cast the sheet in a mold and left then in a vacuum oven to remove bubbles at T_{room} for 24 hours.

3.4 Characterization

3.4.1 Fourier transform infared spectrophotometer (FTIR)

This technique was used to identify absorption mode with 32 scans and a resolution of ± 4 cm^{-1}, wavenumbers range of 4000-400 cm^{-1}, using a deuterated triglycine sulfate as a detector.

3.4.2 The Thermogravimetry Analysis

The thermal behavior of polymers was investigated by weighting a powder sample of 5-10 mg and placed it in a platinum pan, and then heated it under nitrogen flow with the heating rate 10 °C/min from 30-800°C.

3.4.3 Scanning Electron Microscope (SEM)

The morphological structure and surface appearance of doped permethylpolyazine/eyhylene-propylene diene elastomer was obtained. The samples were cut into a small piece and stick on a brass-stub and coated with thin layer of silver.

3.4.4 Electrorheological Property Measurements

Because the change in apparent viscosity is dependent on the applied electric field. This technique determining storage modulus($G'(\omega)$) and loss modulus ($G''(\omega)$) as a function of frequency. The film was cut into disk with a 25 mm diameter and 1 mm thickness.

The storage modulus(G′(ω)) and loss modulus (G″(ω)) were measured as a function of electric field strength, and temperature of samples that varies a small amount of polyazine in range of 5-20%.

3.4.5 The electrical conductivity (σ)

The electrical conductivity was measured by using the two-point probe mater connected with a voltage supplier (Keithley, 6517A) whose constant voltage was varied and the resulatant current measured. The conductivity measurement was performed under the atmospheric pressure, 40-60 %RH and at 25-27°C. The regime where the resultant current is linearly proportional to the applied voltage is called the linear Ohmic regime. The voltage and the current in the regime were converted to the electrical conductivity by following equation:

$$\sigma = 1/\rho = 1/(R \times t) = 1/(R_s \times V \times t)$$

where σ is the specific conductivity (S/cm), ρ is the specific resistivity (Ω.cm), R_s is the sheet resistance (Ω/sq), t is the thickness of sample pellet (cm), V is the applied voltage (Voltage drop) (V), I is the measured current (A), and K is the geometric correction factor of the two-point probe meter. All sample thicknesses were measured by using a thickness gauge.