

CHAPTER III EXPERIMENTAL



3.1 Materials

4-Hydroxybenzoic acid, adenine, *p*-toluenesulfonyl chloride, and sodium sulfate anhydrous were purchased from Fluka Chemicals (Buchs, Switzerland). Paraformaldehyde, cyclohexylamine, potassium carbonate, silica gel and TLC aluminium sheets silica gel 60 were from Merck (Darmstadt, Germany). 1,4-Dioxane, chloroform, toluene, isopropanol, sulfuric acid and sodium hydroxide were purchased from Lab-Scan (Ireland). Dichloromethane and ethanol were purchased from Carlo Erba (Spain). *N,N'*-dimethylacetamide was purchased from Acros (USA). Tetrahydrofuran was from J.T. Baker (USA). Deuterated chloroform was from Aldrich (USA). All chemicals were used AR grade and used without further purification.

3.2 Measurements

Fourier transform infrared (FTIR) spectra were taken at a resolution 4 cm^{-1} by using a Bruker Equinox55/S spectrophotometer equipped with deuterated triglycine (DTGS) detector. Proton nuclear magnetic resonance (^1H NMR) spectra were obtained from a Varian Mercury-400BB spectrometer. The ^1H NMR chemical shifts (δ) are expressed in parts per million (ppm) relative to the proton form of the solvent used. Mass spectra were obtained using a VG Autospec model 7070R from Fison Instruments with VG data system. Samples were run in the positive fast atomic bombardment (FAB(+))MS mode using glycerol as a matrix. Cesium gun was used as an initiator and cesium iodide (CsI) was used as a reference. Elemental analysis (EA) was performed by using a Perkin-Elmer 2400 Series II CHNS/O analyzer with the combustion temperature of 975°C and reduction temperature of 500°C .

3.3 Methodology

The work involves with the details as follow; molecular design, synthesis and characterization of polyamide functionalized with imidazole group. This includes the following plan as shown in Scheme I (page 37),

- Part I*
- Synthesis of aza-methylene phenol derivatives from the ring opening 4-hydroxybenzoic acid based benzoxazine monomer
 - Functionalization of imidazole group at phenol ring

3.3.1 Preparation of 4-hydroxybenzoic acid based benzoxazine monomer

4-Hydroxybenzoic acid based benzoxazine monomer was prepared by the reaction as reported by Ning and Ishida (1994). Carboxylic acid group of the monomer obtained was esterified and the products obtained were structural characterized by FTIR, ^1H NMR, MS, and EA.

3.3.2 Preparation of 4-hydroxybenzoic acid based aza-methylene phenol

4-Hydroxybenzoic acid based aza-methylene phenol was obtained from the ring opening reaction of 4-hydroxybenzoic acid based benzoxazine monomer with ethyl-4-hydroxybenzoate in stoichiometric ratio (1:1). The products obtained were characterized by FTIR, ^1H NMR, MS, and EA.

3.3.3 Functionalization of imidazole group onto 4-hydroxybenzoic acid based aza-methylene phenol

Adenine was chosen as a group to provide imidazole unit. The aza-methylene phenol derivatives obtained was conjugated with adenine. In this step, tosylation of aza-methylene phenol was prepared as a reactive ester species. The functionalization of adenine with aza-methylene phenol was followed by conjugating adenine with tosylated aza-methylene phenol. The products obtained were characterized by FTIR, ^1H NMR, MS, and EA.