

CHAPTER II

EXPERIMENTAL SECTION

2.1 Materials

Polycarbonate grade bisphenol-A was kindly supplied by Siam Chemical Industrial Co., Ltd. Chemical reagents were purchased from the company as follows, and used without further purification. 1,4 - Dioxane, and sodium hydroxide, potassium nitrate, chloroform and picric acid were purchased from Ajax chemicals (Australia); formaldehyde (37% in water) and diethyl ether, anhydrous, from J.T. Baker Inc. (Phillipsburg, USA.); methylamine (40% in water) from Fluka Chemicals (Buchs, Switzerland); sodium sulfate, anhydrous, Nickel (II) nitrate, magnesium sulfate, sodium chloride, toluene (99.5%) and methylene chloride from Farmitalia Carlo Erba (Barcelona, Spain); lithium chloride from Riedel-de Haen (Germany); and calcium chloride was purchased from E. Merck (Germany).

All solvents were stocked with molecular sieve 4 Å to eliminate most of water impurity before used.

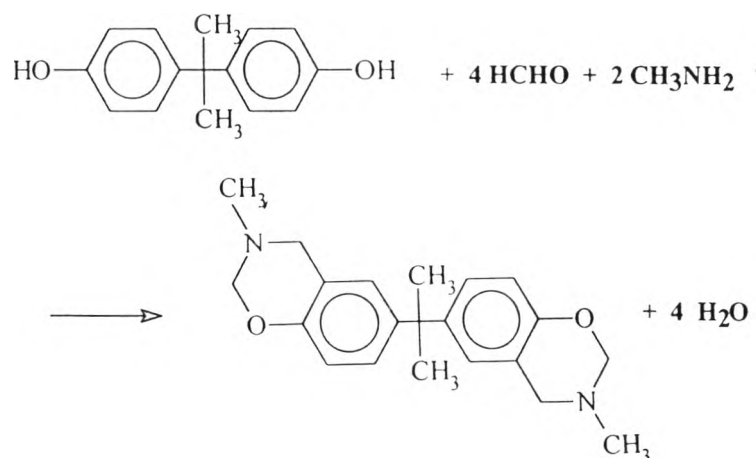
2.2 Material Characterization

B-m precursor and oligomer structures were studied by Fourier transform infrared spectroscopy (FTIR : FT-45A, BIORAD) at a resolution of 8 cm⁻¹. Picrate metal ion concentrations in aqueous phase were measured by

ultraviolet-visible spectroscopy (UV-VIS : Lambda-16, Perkin-Elmer) with scan speed of 240 nm/min, at room temperature. Vortex mixer (GENIE-2, Scientific Industries) was applied for vigorously shake the mixture of organic and aqueous solution for 1 min. The centrifuge (Z 230 A, Hermle) was used for clear separation of a mixture to organic and aqueous phase at 3,000 rpm for 3 min.

2.3 Preparation of Benzoxazine

Bisphenol-A and methylamine-based benzoxazine (B-m) precursor was prepared according to Ning et al. as shown in the following scheme :



Benzoxazine precursor was cured under vacuum at 100°C for 4 hr. As B-m was heated, light yellow oligobenzoxazine was obtained as a thin film. The film was then ground to powder and kept in a refrigerator until use.

2.4 Preparation of Ion Solution

Alkali, alkaline earth and other metal ion solution were prepared by dissolving the corresponding metallic salts in water. 10^{-2} M of picric acid and 2×10^{-2} M of ion salt were dissolved in deionized water. Each ion stock solution was adjusted till the absorbance of wavelength at 354 nm equals to 1. 10^{-1} , 10^{-2} and 10^{-3} g of monomer and oligomer precursors were dissolved in 5 ml of organic solvents, which are methylene chloride, chloroform and toluene.

2.5 Ion Extraction Observation

In this work, benzoxazine is focused on the property of ionophore as a host compound. Ion extraction phenomena was observed according to Pedersen's technique. Five ml. of ionophore in organic solution and 5 ml. of ion solution were mixed vigorously for 3 min., followed by centrifuge for 1 min. The ion concentration in aqueous phase was determined by UV-VIS spectrophotometer at 354 nm. Ion extraction was operated at room temperature by varying the organic phase for various benzoxazine and metal ion species concentrations.

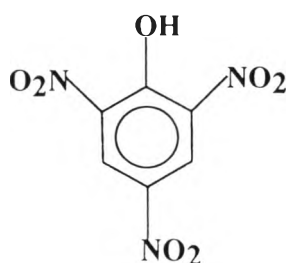


Fig. 5 Picric acid.