

CHAPTER 3 EXPERIMENTAL TECHNIQUE

3.1 The Samples

Copper(II) benzoate and its derivatives were provided by Associates Professor Dr. Norbani Abdullah. They were prepared by the following method.

Copper(II) acetate monohydrate and benzoic acid in a molar ratio 1:2 were refluxed for 24 hours in acetonitrile in a round-bottomed flask connected to a reflux condenser. The mixture was stirred using a magnetic stirring bar. The reaction mixture was left to cool to room temperature and the powder obtained was filtered, rinsed with ethanol and dried in an oven at 60 °C for 30 minutes. The powder was purified by heating with ethanol for 30 minutes. The solid residue was filtered, washed with ethanol and dried in the oven at 60 °C for 30 minutes.

Copper(II) 4-fluorobenzoate, copper(II) 4-chlorobenzoate, copper(II) 4-bromobenzoate, copper(II) 4-iodobenzoate, copper(II) pentafluorobenzoate, copper(II) 4-nitrobenzoate and copper(II) 3,5-dinitrobenzoate were similarly prepared from the corresponding acids as given in Table 3.1.

Derivative	Corresponding Acid
Copper(II) 4-fluorobenzoate	4-fluorobenzoic acid
Copper(II) 4-chlorobenzoate	4-chlorobenzoic acid
Copper(II) 4-bromobenzoate	4-bromobenzoic acid
Copper(II) 4-iodobenzoate	4-iodobenzoic acid
Copper(II) pentafluorobenzoate	pentafluorobenzoic acid
Copper(II) 4-nitrobenzoate	4-nitrobenzoic acid
Copper(II) 3,5-dinitrobenzoate	3,5-dinitrobenzoic acid

Table 3.1 Derivatives of copper(II) benzoate and the corresponding acids

3.2 Thermogravimetric Analysis

The instrument used is Rheometric Scientific Instrument. The sample (3-5 mg) was placed in a cylindrical platinum crucible and heated from 40 °C – 800 °C at a rate of 10 °C/min under nitrogen gas purged at the rate of 24 ml/min.

3.3 Differential Scanning Calorimetry

The weight of the samples (approximately 10 mg) was recorded by microbalance. The samples was placed in an aluminium sample pan and put inside the DSC Rheometric Scientific Instrument. The temperature range is 35 °C to 300 °C with scan rate of 10 °C/min . The samples was purged by nitrogen gas at the flow rate of 10 ml/min.