

## CHAPTER III

### EXPERIMENTAL

#### 3.1 Equipment and Chemicals

##### 3.1.1 Equipment

###### X-ray diffractometer (XRD)

Calcined fibers were analyzed the formation of phase including metal oxide phase or spinel structure phase by XRD (Rigaku / DMAX 2200 HV), using a scan rate of 0.02 theta/min from 10 to 80 2 $\theta$  range. The samples were ground and packed on a glass plate.

###### Scanning electron microscope (SEM)

Both precalcined and calcined fibers were analyzed the morphology and the fiber formation using Hitachi/S-4800 SEM equipped with SE function, at 2 kV voltage, 10  $\mu$ A, and 2000 – 20000x magnitude. Small amount of sample was attached on a carbon tape on the stub.

###### Surface analyzer

Calcined fibers were analyzed the surface area and compared to non-fibers using surface area analyzer (Quantachrome/Autosorb-1) set up function to 24 point calculation, including adsorption, desorption, single point, pore volume and pore size distribution. Before analysis, a sample tube was outgassed for 30 min. Dried samples were then added into the tube, followed by outgassing for 10 – 16 h, and then analyzed

###### Electrospinner

Mixed metal solution was mixed with 10%PVA at 1:10 ratio and spun into fibers by an electrospinner at varied distances between needle and aluminium foil (15 – 20 cm) and varied voltages (12 – 20 kV). The mixture was spun for various times (15 min to 3 h) into fibers collected on the aluminium foil.

### Inductance Capacitance and Resistance (LCR) Meter

Mixed metal oxide fibers at various calcination temperatures were measured conductivity on Agilent E4980A LCR meter that was adjusted frequency and voltage at 50 Hz and 4 V, respectively. The mixed metal oxide powders were compressed as a plate before measuring.

### Furnace

The spun fibers were calcined to eliminate PVA and hydrocarbons by furnace (Lindberg/Blue and Carbolite) at various temperatures (800° – 1100 °C) with a heating rate of 0.3 – 1 °C per min for various heating times (5 – 60 h).

### 3.1.2 Chemicals

Indium acetate ( $\text{In}(\text{CH}_3\text{COO})_3$ , 99.99%), copper acetate ( $\text{Cu}(\text{CH}_3\text{COO})_2$ , 98+%), tin chloride hydrate ( $\text{SnCl}_2 \cdot x\text{H}_2\text{O}$ , 98%), anhydrous citric acid ( $\text{C}_6\text{H}_8\text{O}_7$ ), and diethanolamine ( $\text{C}_4\text{H}_{11}\text{NO}_2$ , 98%) were purchased from Sigma-Aldrich Chemicals Co. Inc., USA. 2-propanol ( $\text{C}_3\text{H}_8\text{O}$ , 99.7%) analytical reagent was purchased from Lab Scan analytical sciences Co. Inc. Ethylene glycol ( $\text{C}_2\text{H}_6\text{O}_2$ , 99.9%) was purchased from J.T.Baker Co. Inc. All chemicals were used without further purification.

## **3.2 Experimental Procedures**

### 3.2.1 Synthesis of $\text{CuInSnO}_4$ by sol-gel method

For indium solution, 0.8 g of indium acetate ( $\text{In}(\text{CH}_3\text{COO})_3$ ) was dissolved in 4 mL of 2-propanol at 85 °C for 1 h, using 0.5 mL of diethanolamine as a stabilizer. For copper solution, copper acetate ( $\text{Cu}(\text{CH}_3\text{COO})_2$ ) 0.5 g was dissolved in 4 mL of 2-propanol, using 1 mL of diethanolamine and 0.5 mL of ethylene glycol as stabilizers. The solution was stirred at room temperature for 30 min. For tin solution, 0.6 g of tin chloride hydrate ( $\text{SnCl}_2 \cdot \text{H}_2\text{O}$ ) was added into 4 mL of 10% citric acid solution and stirred at room temperature for 30 min. Three mixtures were mixed together and stirred at room temperature for 24 h to form viscous solution (*Fu L.J. et al, 2005 and Niasari M. S. et al, 2009*).

### 3.2.2 CuInSnO<sub>4</sub> nanofibers via electrospinning method

Mixed metal mixture was mixed with 10%PVA at 1:10 ratio, which provided a suitable viscosity and less charging, and spun into fibers by the electrospinner at various distances between needle and aluminium foil (15 – 20 cm) and various voltages (12 – 20 kV). The fibers were then calcined at 800–1200 °C to obtain CuInSnO<sub>4</sub> product.