

## CHAPTER 3

### MATERIALS AND METHODS

#### 3.1 INTRODUCTION

This chapter explains research methodology adopted in this study. Briefly, synthesis of SnO<sub>2</sub> NPs and one dimensional nanostructures such as porous nanofibers (PNFs) and multiporous nanofibers (MPNFs) via hydrothermal and electrospinning process, respectively, and also their composites with TiO<sub>2</sub> nanostructures in order to achieve the objectives accomplished during this study. Various techniques are used to study the characteristics of NPs, PNFs, and MPNFs such as XRD for the crystal structure analysis, gas adsorption studies for specific surface area, FESEM and TEM for surface morphology. In addition, procedures to fabricate the DSSCs from the various nano morphologies and details of optical, electrical, and EIS techniques to characterize them are also explained in detail.

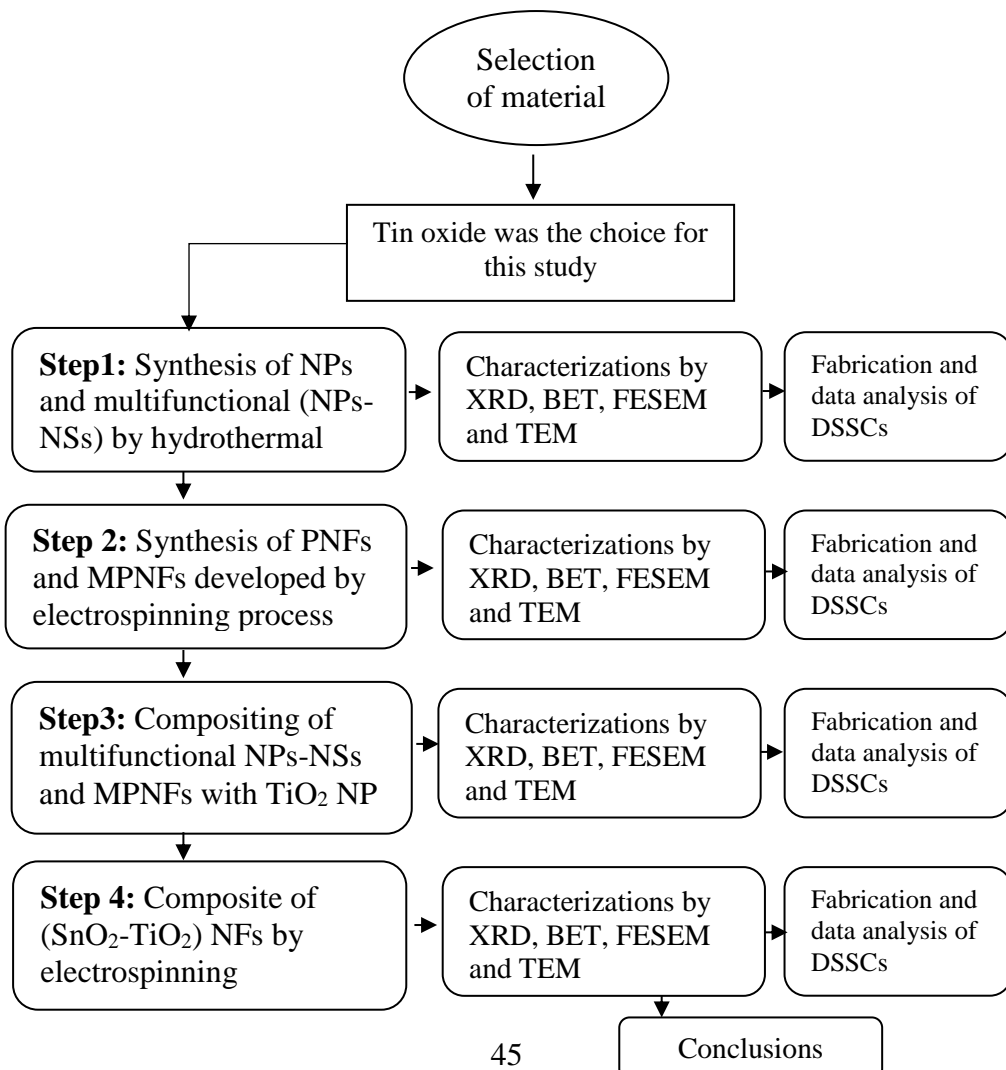
#### 3.2 RESEARCH METHODOLOGY

The flow chart of the research methodology is shown in the schematic 1. At first, suitable MOS materials and their desired morphologies were selected from the literature for the photoanode materials in DSSCs. The SnO<sub>2</sub> was selected as a WE material owing to its higher bandgap (~3.6 eV) and  $\mu_e$  (~100-250 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>). The SnO<sub>2</sub> NPs and multifunctional NPs-nanospheres (NPs-NSs) were the first desired

morphologies to yield high specific surface area and light harvesting properties, which were synthesized using hydrothermal method. The developed materials were employed as a photoanode material in DSSCs. Subsequently, 1D nanostructures (PNFs and MPNFs) were synthesized using electrospinning technique followed by their characterizations and testing as a WE material in DSSCs. Although the multifunctional (NPs-NSs) and MPNFs performed tremendously, the PV parameters still lower than the most employed photoanode  $\text{TiO}_2$ . To improve the PV performance of DSSCs made by these pure nano morphologies, the multifunctional (NPs-NSs) and MPNFs were post-treated so as to form  $\text{TiO}_2$ - $\text{SnO}_2$  composite. Finally,  $(\text{SnO}_2\text{-TiO}_2)$  NFs composite is formed by electrospinning to overcome the limitation of previously formed composites. The PV performance of these pure and composite nanomaterials as a WE material in DSSCs was investigated via their electrical, IPCE, OCVD and EIS characterizations.

Schematic 1

Flow chart of the research activities



### 3.3 SYNTHESIS OF TIN OXIDE NANOSTRUCTURES

In this study, two methods were adopted, viz. hydrothermal and electrospinning, in order to develop various SnO<sub>2</sub> nanostructures. The hydrothermal method was used to synthesize NPs and multifunctional (NPs-NSs) while the electrospinning techniques was employed to produce 1D (PNFs and MPNFs) nanostructures and nanocomposite.

#### 3.3.1 Nanoparticle and Nanospheres Via Hydrothermal

High surface area SnO<sub>2</sub> NPs and multifunctional (NPs-NSs) were synthesized using hydrothermal process by optimizing the reaction temperature and pressure. A typical hydrothermal apparatus is shown in **Figure 3.1**. The crystal nucleation and growth occur in a “Teflon” container. The polymer required to have a higher melting point than the reaction temperature in order to avoid any impurity during the processing time.

The size and morphology of the nanostructures formed by this method depend on various parameters such as pressure inside the Teflon, operating temperature, precursor constituents and reaction time. Careful observation and precaution should be kept in mind during the experiment as follows: (i) the solution volume should not exceed 80% than the total volume of the Teflon in order to allow sufficient space for the liquid swelling during reaction time and (ii) the temperature of the reaction should be increased or decreased with a ramping rate of less than 5 °C/min to avoid any thermal shock. In our experiment, SnO<sub>2</sub> NPs and its multifunctional NPs-NSs were synthesized following a reported method (Wang et al., 2012) with modifications. However, the concentration of the growth solution was kept (16.5 mM) nearly one half than that reported before (32.7 mM) (Wang et al., 2012). In a typical procedure, SnCl<sub>2</sub> · 2H<sub>2</sub>O (0.25 g) was mixed with 1 N HCl (0.6 mL), ethanol (6 mL) and DI water (60 mL) and stirred in the sonication bath for an hour. The resultant transparent solution was transferred to an autoclave and kept at a pre-heated furnace at ~150 °C for ~24 h. After cooling to the room temperature, the solution was centrifuged, washed three times with DI water. After the cleaning process, the sample was dried in the oven at ~70 °C for overnight. This sample was labelled as “sample 150 °C”. The same procedure was adopted for “sample 180 °C” and “sample 200 °C” with increase in the furnace temperature to ~180 °C and ~200 °C, respectively.