CHAPTER 3

EXPERIMENTAL METHODS

3.1 INTRODUCTION

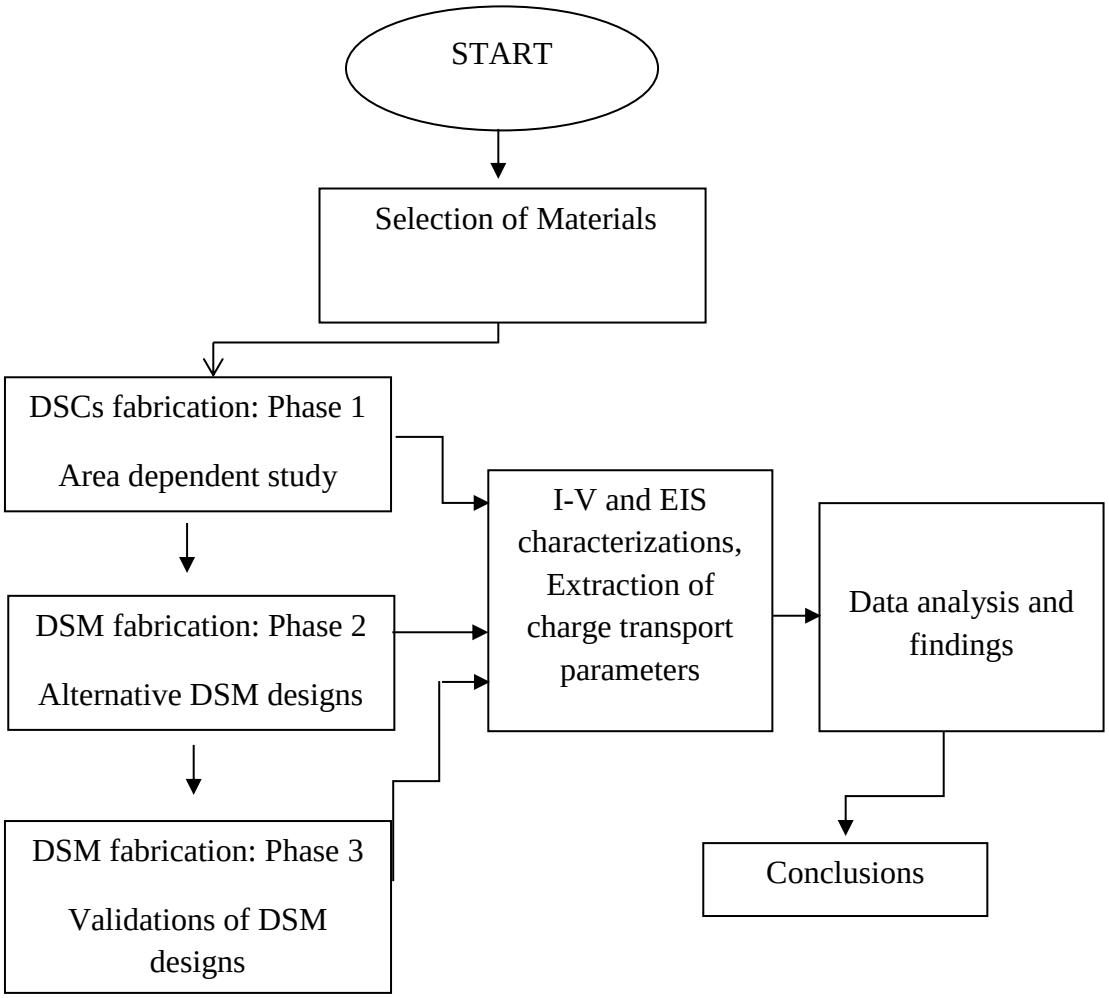
This chapter highlights the research methodology as well as various materials and methods used in the present research to achieve the objectives. Various experimental designs for DSCs fabrication, the tools and techniques of characterizations, the possible errors, the precautions and correction made, and also the extraction mechanism of various charge transport parameters to identify the kinetics of the DSCs are described in this chapter.

3.2 RESEARCH METHODOLOGY

The methodology adopted to achieve the objectives of this research is summarized in flow chart 3.1. 3

The procedure starts by fabricating DSCs with varying areas while keeping other device physical parameters unchanged. The results are analyzed to develop novel device designs to offer improved charge collection efficiency.

Flow chart of research activities



3.3 MATERIALS

Unless stated otherwise, the widely employed commercial DSC materials were used for fabrication of DSCs throughout this study. The solvents used in this study, viz., ethanol, acetone, hydrochloric acid (HCl), and titanium chloride ($TiCl_4$) were purchased from Sigma Alrdrich. Commercially available TiO_2 paste (Solaronix Ltd.) was employed as the mesoporous photoanode layer. The substrates (FTO), cis-bis(isothiocyanato)bis(2,2'-bipyridyl-4,4'-dicarboxylato)-ruthenium(II) (N3 dye), iodide/triiodide based redox electrolyte, platinum coated counter electrode , and surylin spacer (30 μm) were purchased from Solaronix Ltd.

3.4 PHOTOVOLTAIC FABRICATION

3.4.1 Phase 1: Area Dependent Photoanodes

The DSCs were fabricated on fluorine doped tin oxide coated glass substrates (FTO) using standard TiO_2 paste, cis-diisothiocyanato-bis(2,2'-bipyridyl-4,4'-dicarboxylic acid) ruthenium(II) (N3 dye), and the iodide/triiodide electrolyte. The FTO substrates were pre-cleaned by 15 min sonication in distilled water, ethanol and acetone. Pre-cleaning of substrates is crucial to remove any organic impurity on the surface of the FTOs which may lower the performance of the DSCs. The substrates were coated with a dense thin hole blocking layer via a 50 mM $TiCl_4$ solution treatment. The solution was made by adding $TiCl_4$ in distilled water at ~0 ^oC and then stirring vigoursly for 15 min. The pre-cleaned substrates were immersed in this solution and kept at 70 ^oC for 30 min to form a dense blocking layer. Eight areas were selected in the range 0.16 – 1.96 cm^2 for the present study. The TiO_2 paste was coated on the active areas by screen printing technique followed by drying at ~100 ^oC for ~2 – 3 h. Thus films were then sintered at an optimized ramping program at 450 ^oC for 30 min. The ramping program was as follows: 2 ^oC/min until 250 ^oC and stay of 10 min and 5 ^oC/min until 450 ^oC and stay of 30 min). We noticed that annealing of the TiO_2 films require intensive precision; crack in TiO_2 films are noticed otherwise. Translucent TiO_2 electrodes thus produced were anchored to the N3 dye by dissolving in a 1:1 volume mixture of acetonitrile and tert-butanol for 24 h at room temperature. The dye-sensitized samples were then washed in ethanol to remove unanchored dye and dried in air. Samples were sealed using a 50 μm spacer (Surilyn). Acetonitrile containing 0.1M lithium iodide, 0.03M iodine, 0.5M 4-*tert*-butylpyridine and 0.6M 1-propyl-2,3-dimethyl imidazolium iodide was used as the electrolyte. A great care is to be given while filling the electrolyte as an improper filling may cause air to be trapped inside the gap which affects the performance of DSCs. A platinum coated FTO glass was used as the counter electrode.