CHAPTER III

MATERIALS AND METHODS

3.1 INTRODUCTION

This chapter details the research methodology which comprises of various materials and methods used in the present research. The details of the synthesis protocol and development of nanowires, the tools and techniques used for characterizations of the materials, the possible errors, the precautions and correction made, preparation and testing of supercapacitor electrodes, and fabrication of asymmetric supercapacitors and testing is elaborated in this chapter.

3.2 RESEARCH METHODOLOGY

Flowchart 3.1 outlines the research methodology adopted in this work. The materials were synthesized by electrospinning technique using an aqueous polymeric solution based electrospinning; the as-spun polymeric fibers were annealed to get ceramic nanowires; the nanowires thus obtained were characterized for its morphology, crystal structure, and surface properties. The supercapacitor electrodes were fabricated using those nanowires and tested their electrochemical properties. Finally asymmetric supercapacitors were fabricated using the nanowires as one of the electrodes and commercial activated carbon as the other electrode; the devices were tested electrochemically. The techniques used for synthesis, characterization, device fabrication, and testing are detailed in this chapter subsequently.

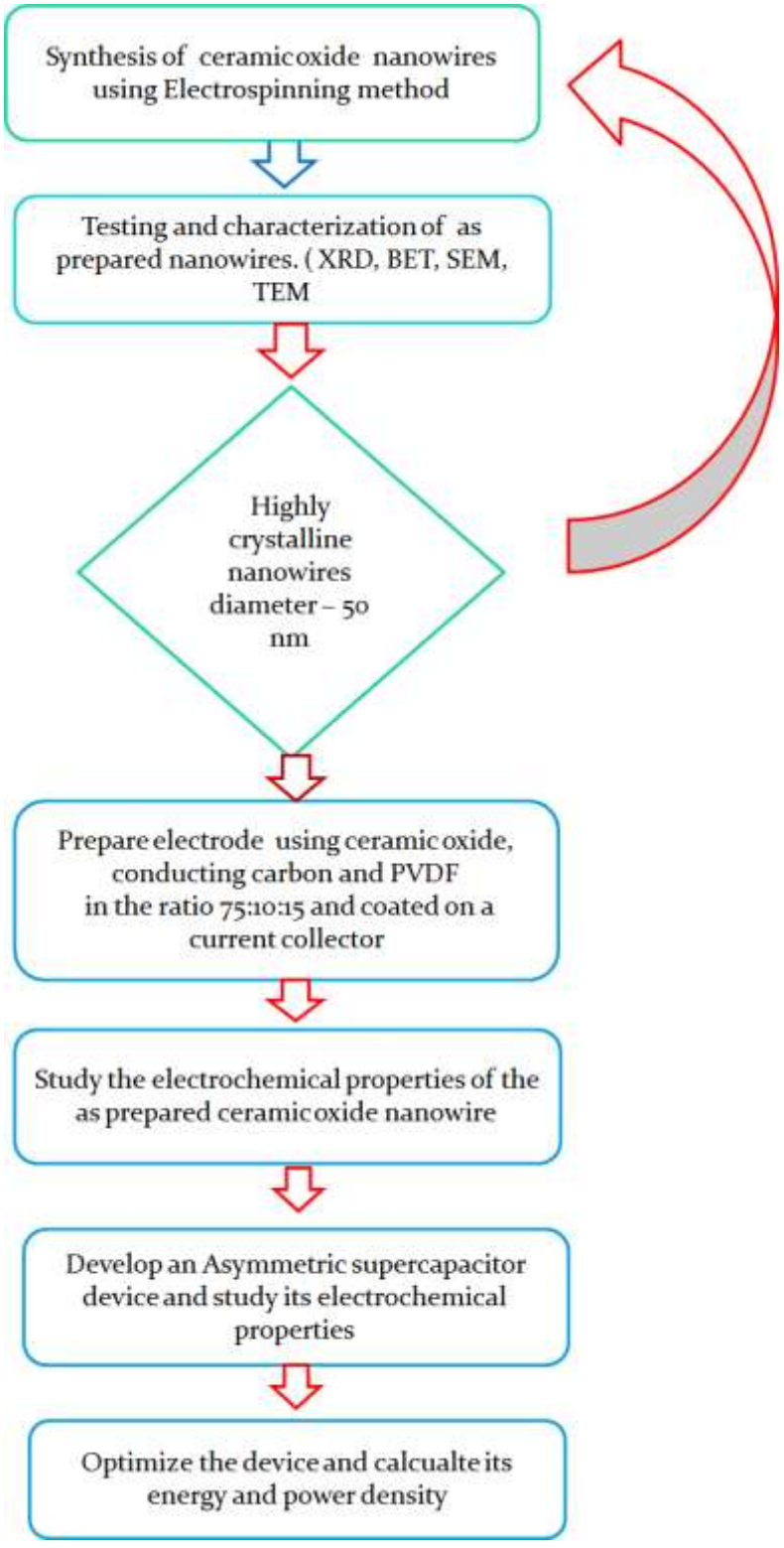

Figure 3.1: Summary of methodology adopted in this work.

3.2 ELECTROSPINNING TECHNIQUE

The electrospinning is a simple technique for fabrication of continuous nanofibers of polymers and polymeric composites (Ramakrishna et al., 2010; Reneker and Yarin, 2008). A typical electrospinning set up consists of a high voltage power supply, a programmable syringe pump and a grounded collector as in Figure 3.2&3.3. During the process, a polymeric solution is injected from a small nozzle in a region of high electric field (~$10^5 Vm^{-1}$). The build-up of electrostatic charge on the surface of a liquid droplet induces the formation of a jet, which is then stretched to form a continuous fibre. The solution evaporates before it reaches the collecting drum and solid fibers are collected.

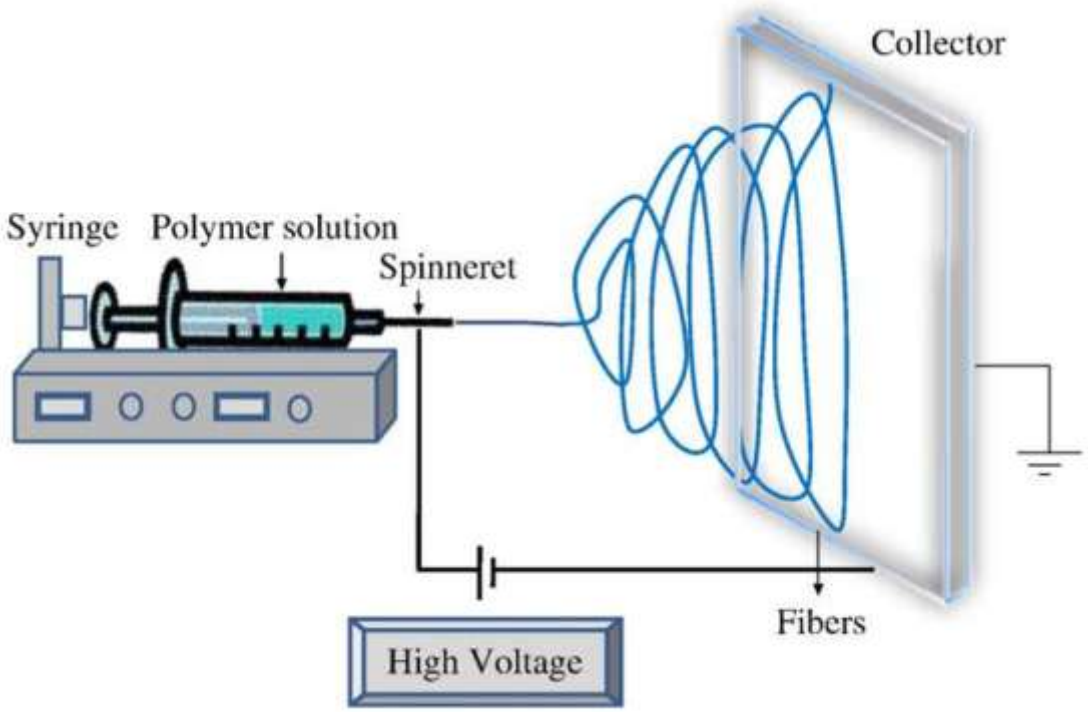

Figure 3.2: Schematic showing electrospinning set up. Adapted from (Bhardwaj and Kundu, 2010).

The electrospinning process is controlled by three parameters, viz. (i) solution parameters, (ii) process parameters, and (iii) ambient parameters. The solution parameters include viscosity, conductivity, molecular weight, and surface tension (Bhardwaj and Kundu, 2010). The process parameters include applied electric field, needle to collector distance, and flow rate. The ambient parameters include humidity