<u>Abstract</u>

The ever-increasing demand for energy encourages us to search for alternate energy sources to restrict the indiscriminate use of fossil fuels. Supercapacitors are crucial candidates in the field of energy storage devices. Despite all the advantages associate with the supercapacitors, they still suffer due to the low energy density delivery. Hence a lot of attention has been conferred towards the enhancement of energy density. Batterytype transition metal chalcogenide electrodes show better electrochemical properties in comparison to conventional capacitors. The broad objective of this thesis is the fabrication and electrochemical evaluation of transition metal chalcogenide-based battery-type electrodes for supercapacitor application. A simple, inexpensive, one-pot and binder-less synthetic technique has been developed. The novel feature of the developed synthetic technique lies in the fact that the nanostructures could be directly developed on Ni foam current collector substrates using the chemical bath deposition technique. Based on the facile chemical bath deposition technique, NiSe-Se nanotubes, CoSe-Se nanotubes, NiO nanoflowers, NiSe-Se nanotubes@NiO nanoflowers, and NiO nanoflowers@ NiSe-Se nanotubes were grown on Ni foam current collector. Subsequently, the synthesized materials were subjected to various electrochemical tests like cyclic voltammetry, galvanostatic charge-discharge, and electrochemical impedance spectroscopy. The novel concept of in-situ selenization has been included in the chemical bath deposition technique which not only reduced the total time of synthetic compared to ex-situ selenization methods but also reduced the cost of fabrication. Novel strategies were incorporated to increase the conductivity as well as the active surface area of the fabricated electrodes. Highly porous nanostructures were developed on current collector surfaces to increase the electro-active. NiSe-Se@Ni foam electrode showed a superior specific capacitance value of 2447.46 F g^{-1} at a current density value of 1 A g^{-1} in 1M aqueous KOH electrolyte. A pouch-type hybrid supercapacitor (HSC) device was fabricated with NiSe-Se@Ni foam//AC@Ni foam delivered a specific capacitance of 84.10 F g⁻¹ at a current density of 4 mA cm⁻² with an energy density of 29.90 W h kg⁻¹ at a power density of 594.46 W kg⁻¹ for an extended operating voltage window of 1.6 V. The fabricated pouch-cell was suitable for real-world applications. A similar synthetic strategy was extended to develop CoSe-Se@Ni foam which exhibited a specific capacitance of 1750.81 F g⁻¹ at 1 A g⁻¹. A pouch-type hybrid supercapacitor (HSC) device was fabricated with CoSe-Se@Ni foam//AC@Ni foam delivered a specific capacitance of a specific capacitance value of 106.73 F g⁻¹ at 0.5 A g⁻¹ and achieved an energy density of 37.94 Wh kg⁻¹ at a power density of 475.30 W kg⁻¹. Finally, a hierarchy-based electrochemical study was conducted with NiSe-Se@NiO@Ni foam and NiO@NiSe-Se@Ni foam electrodes. The electrodes exhibit superior specific capacitance of 2704.92 F g⁻¹ and 1891.50 F g⁻¹ at a scan rate of 5 mV s⁻¹, respectively, which is 3.25 and 2.27 times higher than that of the value of pristine NiO nanoflowers (831.50 F g^{-1}). The study further showed the synergistic contribution of electro-active materials present in the sample. A sustainable approach was conducted to reutilize the used electrodes. The

waste electrodes were collected, upcycled, and tested against *E. coli* bacteria for antibacterial application.

Keywords: battery-type, hybrid supercapacitor, NiSe, NiO, CoSe, Ni foam, E-waste, NiSe-Se@NiO@Ni foam, NiO@ NiSe-Se @Ni foam, pouch cell, electrode