CHAPTER III
AIM AND SCOPE OF THE WORK

3.1 Scope of the work

Recent research is focused on the chitosan based metal / metal oxide composites because of its mechanical and metal-binding properties [172-176]. Various types of chitosan-metal oxide composites were prepared for different applications such as chitosan-TiO$_2$ nanocomposite membranes for ethanol dehydration [177]. TiO$_2$-chitosan/glass photocatalyst was used as removing reagent for the removal of monoazo dye via photodegradation-adsorption process [178], fluorescent chitosan-ZnO nanoparticles were used as biological fluorescent labeling and a carrier for guest materials [179], chitosan-ZnO nanoparticles composite film deposited on the surface of platinum electrode for determination of TG [180], chitosan/nano-ZnO composite membranes for antibacterial and mechanical properties [11] and chitosan–zinc oxide nanoparticles for dye adsorption [12].

All the reported chitosan based metal / metal oxide composites were prepared using chitosan as base material and prepared metal oxide nanoparticles as fillers. Generally, chitosan was prepared from chitin by heating with 40-50% of NaOH solution [181] and ZnO was prepared from zinc source material where NaOH playing as a precipitating agent [182]. There is no literature
available for the combination of chitin, zinc salt and NaOH in a single bath process for preparation of chitosan-ZnO nanocomposites.

3.2 Aim of the present work

The aim of the present work were confined

- to prepare chitosan-ZnO nanostructures in single step
- to find effective zinc chloride concentration
- to find out the effect of NaOH concentration and its addition
- to study the effect of annealing
- to determine the dye adsorption efficiency
- to incorporate graphene oxide with chitosan-ZnO in single step
- to study the optical and electrical conductivity

3.3 Methodology of the work

- Chitin was extracted from the sea waste of crab shells
- The extracted chitin was treated with different percentage of zinc chloride and sodium hydroxide solution.
- As-prepared chitosan-ZnO nanostructure was annealed at different temperature
- Application of chitosan-ZnO composites on chromium complex dye
- In the chitosan-ZnO preparation process, graphene oxide solution was added to prepare graphene-ZnO/chitosan
Characterization of prepared samples for

Functional group identification by Fourier Transform Infra-Red Spectroscopy (FTIR)

Crystallite size and phase identification by X-Ray Diffraction (XRD)

UV absorption by Ultraviolet Visible Spectroscopy (UV-Vis)

UV emission by Photoluminescence Spectroscopy (PL)

Surface morphology by Scanning Electron Microscopy (SEM)

Determination of particles size by Transmission electron microscopy (TEM)

Metal and oxygen content by Energy Dispersive X-ray Analysis (EDAX)

Electrochemical performance by cyclic voltammetry (CV) and Impedance spectroscopy (IS)

Surface morphology, thickness and lateral size of graphene oxide sheets by Atomic Force Microscopy (AFM)

Graphene oxide D line and G line peaks by Raman Spectroscopy

Electrical conductivity of graphene oxide by Four-probe resistivity method (Keithly)

Graphene oxide conductivity by Current-Voltage (I-V) measurement
3.4 Experimental investigation on chitosan-ZnO nanostructures and its graphene oxide hybrid composites

A search of literature revealed that only a considerable amount of work has been reported on preparation of chitosan-ZnO and chitosan-graphene oxide from the chitosan polymer with the addition of already prepared metal oxide nanoparticles/ graphene oxide. But, there are no reports on preparation of chitosan-ZnO nanostructures and chitosan based graphene oxide-ZnO hybrid composite using single bath. An experimental investigation of chitosan-ZnO complex was prepared during chitin deacetylation. The extracted chitin dissolved in acetic acid with addition of zinc chloride solution and then bulk addition of already prepared aqueous sodium hydroxide solution. In order to improve the optical property of chitosan-ZnO nanostructure was prepared using chitin dissolved in acetic acid with different concentration of zinc chloride solution and then micro-addition of NaOH solution.

Novel chitin and chitosan based graphene oxide-ZnO hybrid composites was prepared using chitin dispersed in zinc chloride solution and synthesized graphene oxide where NaOH solution was added by micro-addition. The prepared hybrid composites electrical conductivity was studied by Impedance, four-probe resistivity and I-V technique. The optical property was studied by UV-vis spectroscopy.