CHAPTER II

SCOPE OF THE PRESENT INVESTIGATION

2.1 Electrochemical sensors

Metal phthalocyanines are the important classes of molecule which are widely used in various fields including solar cells [108], electrocatalyst [109] and chemical sensor [110]. In case of water soluble forms of MTsPc have been used as dopant as well as catalyst for the various electron transfer oxidation process. Metal phthalocyanine or substituted metal phthalocyanine modified electrodes have been utilized for the electrochemical oxygen reduction reaction [111]. Electrocatalytic oxidations of some biologically important thiol detection of toxic pollutants such as nitrite, sulphite, hydrazine, hydrogen peroxide, amitrol, etc are some of the recent evidence. Also, it can be used as mediator for the detection of dopamine and ascorbic acid. Because of the potential application of water soluble sulphonated metal phthalocyanine, we have taken challenge to develop method for the synthesis of tetrasulphonated metal phthalocyanine as dopant to incorporate into the polyaniline matrix by an interfacial polymerization. Since the nanocomposites consist of three components to like tetrasulphonated metal phthalocyanine, conducting polyaniline and silver nanoparticles, we believe that the conductivity being increased in many folds and the surface area of the system also increases. From the interfacial polymerization method, it is possible to develop nanotubular structure of polyaniline. These properties are important characteristics for the development of electrochemical sensors [112]. In the present investigation the following methodology was adapted to carry out a systematic approach for the
preparation of nanostructure material which contains a three component system as mentioned in the earlier paragraph.

**Objectives of the present work**

Synthesis of MT$_2$Pc doped PANI decorated Ag NP’s by Interfacial polymerization method and study their electrocatalytic application. [M = Cu, Ni, Co and Fe]

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**Fig. 2.1  Flow chart of the present work**

2.2 Objectives

Recently, we have established a single pot method for the synthesis of polyaniline protected silver nanoparticles by chemical oxidative polymerization by biphasic methods in which the aniline was allowed to react with metal salts (AgNO$_3$) under static condition at room temperature (113). The proposed method is one of simple way of synthesis of conducting polymer protected metal nanoparticles for various electrocatalytic applications. We have also established the oxidative polymerization of aniline and its derivatives were used for synthesis of polyaniline and substituted polyaniline protected metal nanoparticles under interfacial polymerization approach (114).
The structure of the p-aminodiphenylamine and water soluble tetrasulfonated metal phthalocyanine used in the present work is shown below;

![Chemical structure of p-aminodiphenylamine](image)

**p-aminodiphenylamine**

![Chemical structure of tetrasulfonated metal phthalocyanine](image)

**Tetrasulfonated metal phthalocyanine [M = Fe, Co, Ni & Cu]**

**Fig. 2.2** Structure of p-aminodiphenylamine reactant and tetrasulfonated metal phthalocyanine dopant
The objectives of the present investigation are given below:

(i) To synthesis MTsPc doped polyaniline using AgNO₃ as oxidizing agent in presence of aniline dimer by interfacial polymerization method under static condition at room temperature in 0.1 M HCl in presence of MTsPc as catalyst.

(ii) To synthesis MTsPc doped polyaniline using (NH₄)₂S₂O₈ as oxidizing agent in presence of aniline dimer under acidic pH in presence of MTsPc as catalyst.

(iii) To characterize the MTsPc doped polyaniline protected AgCl nanoparticles using various instrumental methods, such, UV-Visible, FT-IR, XRD, TGA, FE-SEM and cyclic voltammetry.

(iv) To study the application of the nanocomposites against oxidation of nitrite (NO₂⁻), hydrazine (NH₂NH₂) and reduction of hydrogen peroxide (H₂O₂) and

(v) To use the nanocomposites for the detection of some inorganic pollutants like nitrite (NO₂⁻), hydrazine (NH₂NH₂) and hydrogen peroxide (H₂O₂) in water samples.

2.3 Present work

The methodology for the synthesis of metal tetrasulfonate phthalocyanine doped polyaniline using p-aminodiphenylamine (dimer aniline) as starting precursor in presence of silver nitrate in 0.1 M HCl in the schematic diagram. The polymerization reaction was carried out by interfacial route to grow polyaniline in a controlled manner.
The synthesis of polyaniline protected silver nanoparticles in presence of water soluble metal phthalocyanine as dopant as well as shape directing agent.

(i) Oxidative polymerization of aniline dimer using a metal salt, AgNO₃ in a phase transfer method and

(ii) In order to confirm the formation of polyaniline protected silver chloride nanoparticles [115] we demonstrated the interfacial polymerization using hydrogen peroxide instead of silver nitrate under identical experimental condition.

The MTsPc doped polyaniline protected silver chloride nanoparticles and MTsPc doped polyaniline have been synthesized by interfacial polymerization method. The resulting solid metal nanoparticle doped polyaniline [116] was collected by centrifugation and then dried under nitrogen atmosphere. Similarly all the four tetrasulfonated metal phthalocyanine [M = Fe, Co, Ni & Cu] doped AgCl nanoparticle containing PANI were synthesized. A systematic characterization studies have been carried out to measure the size and shape, thermal stability and their optical and electrochemical redox properties. Finally, the electroanalytical technique was used for the detection of nitrite, hydrazine and hydrogen peroxide in low concentration ranges.