CONTENTS

CHAPTER II

SYNTHESIS TECHNIQUES

CHAPTER III

SYNTHESIS AND CHARACTERIZATION OF β-CYCLODEXTRIN FUNCTIONALIZED REDUCED GRAPHENE OXIDE NANOSHEETS FOR ELECTROCHEMICAL DETECTION OF NITROPHENOL

CHAPTER IV

INVESTIGATION ON DECORATION OF SILVER NANOPARTICLES ON POLYMER FUNCTIONALIZED REDUCED GRAPHENE OXIDE NANOSHEETS FOR ELECTROCHEMICAL SENSING APPLICATIONS

CHAPTER V

INFLUENCE OF GOLD NANOPARTICLES CONCENTRATION ON POLYMER FUNCTIONALIZED REDUCED GRAPHENE OXIDE NANOSHEETS AND ITS ELECTROCHEMICAL SENSING PERFORMANCE

CHAPTER VI

IMPACT OF STRUCTURAL AND MORPHOLOGIAL PROPERTIES OF rGONS/β-CD/ZnO NANOCOMPOSITES ON THE ELECTROCHEMICAL DETECTION APPLICATION

CHAPTER VII

INCLUSION OF COPPER OXIDE NANOPARTICLES AND β-CD POLYMER FOR THE ENHANCEMENT OF ELECTROCATALYTIC PROPERTY OF REDUCED GRAPHENE OXIDE NANOSHEETS AND ITS SENSING APPLICATIONS

CHAPTER VIII

SUMMARY OF RESULTS AND CONCLUSION

ABSTRACT

The present research work mainly focuses on the development of efficient electrode modified material using various concentrations of metal (Au, Ag) / metal oxide (ZnO, CuO) nanoparticles embellished β-cyclodextrin functionalized reduced graphene oxide nanosheets for the effective electrochemical detection of nitrophenol isomers (ortho-, para- and meta-nitrophenol). The synthesized nanocomposites are analyzed through various characterization techniques to investigate their functional, structural and morphological properties along with their electrocatalytic applications.

Graphene oxide (GO) nanosheets are prepared by modified Hummer's method and functionalized using β-cyclodextrin polymer (rGONS/β-CD) via chemical reduction method. The influences of β-cyclodextrin polymer on the functional, structural and morphological properties of reduced graphene oxide nanosheets are investigated using Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), Scanning electron microscopy (SEM), Energy dispersive X-ray analysis (EDAX) and High resolution transmission electron microscopy (HRTEM) with Surface area electron diffraction (SAED) analysis. The XRD pattern of GO and rGONS/β-CD nanocomposites indicate the formation of GO and polymer functionalized reduced graphene oxide nanosheets with the sharp and broad diffraction peak at 10.3˚ and 23˚ respectively. The SEM analysis showed the formation of wrinkled and less transparent β-CD functionalized reduced graphene oxide nanosheets. The elemental compositions of the synthesized samples are characterized using EDAX analysis and confirmed the presence of carbon and oxygen elements without any impurities. The electrocatalytic analysis is performed for the detection of ortho-nitrophenol isomer using GO, rGONS and rGONS/β-CD nanocomposite modified GCE and the results show a well defined redox peak with the current value of -0.21 mA for rGONS/β-CD nanocomposite modified GCE than the other nanocomposite modified electrodes.

The various concentrations of silver nanoparticles embellished β-cyclodextrin functionalized reduced graphene oxide nanosheets are synthesized by simple wet chemical method. The influence of various concentrations of silver nanoparticles on the structural, morphological and electrocatalytic properties of rGO/β-CD nanosheets are studied using FT-IR, XRD, SEM, EDAX and TEM with SAED analysis. It is observed

from XRD analysis that the crystallite size of the silver nanoparticles varied between 18 nm to 23 nm. TEM analysis shows the formation of spherical shaped silver nanoparticles in the rGO/β-CD nanosheets and the SAED pattern confirms its polycrystalline nature. The electrochemical analysis revealed that the synthesized rGONS/β-CD/Ag nanocomposite is best suited for the detection of ortho-nitrophenol with the linear range of detection from 1 mM to 12 mM and sensitivity value of about 0.28 mA mM^{-1} cm^{-2}.

The different concentrations (0.002 M, 0.004 M, 0.006 M, 0.008 M and 0.01 M) of gold nanoparticles encapsulated β-cyclodextrin functionalized graphene oxide nanocomposites (GO-β-CD-Au) are synthesized via wet chemical method and the effect of gold nanoparticles concentrations on the surface property of rGONS/*β*-CD nanocomposite is investigated using FT-IR, XRD, SEM, EDAX and TEM analysis. The FT-IR spectra reveal that the blue shift in the bands may be due to the intermolecular interaction between the graphene oxide and β-cyclodextrin. The structural analysis reveals that the crystallite size of the gold nanoparticles ranges from 9.16 nm to 9.20 nm for the concentration of 0.002 M to 0.006 M and there is an increase in the crystallite size from 9 to 12 nm for 0.008 M and 0.01 M. This may be due to the aggregation of gold nanoparticles encapsulated on the surface of rGONS/β-CD. The SEM analysis shows that the rGONS/β-CD/Au nanocomposite synthesized using 0.006 M concentration of gold (III) chloride trihydrate is uniformly decorated with large number of gold nanoparticles. The TEM analysis confirms the formation of uniformly decorated spherical shaped gold nanoparticles on the surface of wrinkled cyclodextrin functionalized reduced graphene oxide nanosheets. The physico-chemical studies revealed the impact of gold nanoparticles concentration on the electrocatalytic property of rGONS/CD nanocomposites. It is further confirmed by the cyclic voltammetry analysis and the results reveals a better linear detection range over the concentration from 100 μM to 280 μM with the sensitivity value of about 8.87 mA$\mu M^{-1}cm^{-2}$ for the detection of ortho-nitrophenol. Hence, the synthesized electrode material is better for the detection of ortho-nitrophenol than para- and meta-nitrophenol isomers.

The facile wet chemical method for the synthesis of novel zinc oxide (ZnO) nanoparticles embellished beta-cyclodextrin (β-CD) polymer functionalized reduced graphene oxide (rGO) nanosheets (rGONS/β-CD/ZnO) using different molar ratios of zinc acetate dihydrate is reported. The synthesized rGONS/β-CD/ZnO nanocomposites

are investigated using FT-IR, XRD, SEM, EDAX and TEM with SAED analytical techniques. The molar ratio of Zn^{2+} ions in the reaction system plays a crucial role on the structural and morphological properties of rGONS/β-CD/ZnO nanocomposite and also in its electrocatalytic applications. The XRD analysis shows that the crystallite size of ZnO nanoparticles varies linearly for the molar ratio of Zn^{2+} ions from 0.002 M to 0.008 M and dramatically increases for the molar ratio of 0.01 M, due to the aggregation of ZnO nanoparticles on the rGONS/β-CD surface. The SEM and TEM morphological analysis reveals that the spindle like zinc oxide nanoparticles is effectively embellished on the rGONS/β-CD surface without agglomeration for the molar ratio of Zn^{2+} ions from 0.002 M to 0.008 M. The electrochemical study shows that the rGONS/β-CD/ZnO nanocomposite synthesized using the molar ratio of Zn^{2+} ions from 0.002 M to 0.008 M exhibits linear detection response with higher electrocatalytic activity towards the detection of nitrophenol isomer, which may be attributed to the large surface area from high loading concentration of ZnO nanoparticles with smaller crystallite size on the rGONS/β-CD surface. The cyclic voltammetric response of 0.008 M molar ratio of synthesized rGONS/β-CD/ZnO nanocomposites shows the best linear range of detection for para-nitrophenol isomer among the three nitrophenol isomers from 10 to 240 μm with a high sensitivity value of 13.8 mA $\mu M^{-1}cm^{-2}$.

The synthesis of beta-cyclodextrin (β-CD) functionalized copper oxide nanoparticles (CuO) encapsulated reduced graphene oxide (rGO) nanosheets (rGONS/β-CD/CuO) for the sensitive electrochemical detection of nitrophenol isomers is described. The functionalization and encapsulation of reduced graphene oxide nanosheets surface using beta-cyclodextrin and copper oxide nanoparticles are done by using chemical reduction of graphene oxide with beta cyclodextrin and copper (II) acetate monohydrate chemical reagents. The physico-chemical properties of synthesized rGONS/β-CD/CuO nanocomposites are investigated using FT-IR, XRD, SEM and TEM analytical techniques. The SEM and TEM morphological analysis confirms that β-CD molecules are effectively functionalized on the surface of reduced graphene oxide nanosheets and also the encapsulated spherical shaped copper oxide nanoparticles on the surface of homogenous rGONS/β-CD nanosheets. The electrochemical study shows that the rGONS/β-CD/CuO nanocomposite modified GCE exhibits a good electrochemical behaviour for the reduction of para-nitrophenol with linear range of

detection 60 μm to 90 μm and 120 μm to 280 μm and the sensitivity of 10.4 $mA\mu M^{-1}cm^{-2}$.

The results of the present work summarize the synthesis of graphene based polymer functionalized meta/metal oxide nanoparticles at nanometer size with the appreciable structural and morphological properties and are best suited for the electrochemical sensor applications.

LIST OF FIGURES

LIST OF TABLES