Studies of Transition Metal Nanoparticles with Tetracyanoquinodimethane for Electrochemical Sensing Application

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Doctor of Philosophy

By

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CHAPTER- 6

Summary and Future Work

6.1 Summary

The thesis entitled, ***"Studies of Transition Metal Nanoparticles with Tetracyanoquinodimethane for Electrochemical Sensing Application"*** described the synthesis of transition metal nanoparticles for electrochemical sensing of ascorbic acid, dopamine, and NADH. The content of the thesis has been divided into the following five chapters.

1. Introduction and Literature Survey.
2. Instrumentation and Experimental Techniques.
3. Effect of palladium and its nanogeometry on the redox electrochemistry of tetracyanoquinodimethane modified electrode; Application in electrochemical sensing of ascorbic acid.
4. Fabrication of dopamine sensor based on a carbon paste electrode modified with tetracyanoquinodimethane and cobalt tungstate nanoparticles.
5. Design of a low voltage TCNQ-Pd-Co@NC modified electrode-based NADH sensor.

Chapter 1 deals with the general idea and basic concepts of biosensors, types of biosensors, characteristics of biosensors, nanomaterials, types of nanomaterials, the application of transition metal nanomaterials in biosensors, the redox mediator, tetracyanoquinodimethane, and benefits of the proposed materials for sensing.

Chapter 2 describes different experimental techniques that have been used for the characterization of synthesized nanomaterials. The main techniques that have been

employed for characterizations are X-ray diffractometer, scanning electron microscope, transmission electron microscope, atomic force microscopy, and X-ray photoelectron spectroscopy, used for morphological and structural investigation. Cyclic voltammetry, differential pulse voltammetry, amperometry, and electrochemical impedance spectroscopy techniques have been used for electrochemical characterization. The flowchart of the research strategy is also discussed.

Chapter 3 In this chapter, we have synthesized two different-sized palladium nanoparticles, mixed them with tetracyanoquinodimethane (TCNQ), and fabricated TCNQ-PdNP-1 and TCNQ-PdNP-2 modified carbon paste electrode. This TCNQ-PdNPs modified carbon paste electrode is electrochemically characterized by cyclic voltammetry, amperometry, differential pulse voltammetry, and electrochemical impedance spectroscopy. The sensitivity and limit of detection of TCNQ-PdNP-2 were calculated by using amperometric analysis and the sensitivity was found to be 14.8 μA mM^{-1} and the limit of detection was 30.35 μM. Real sample analysis was performed by using the standard addition method taking an ascorbic acid tablet sample solution.

Chapter 4 In this chapter, we have synthesized $CoWO_4$ nanoparticles by hydrothermal method, and the nanoparticle was characterized by scanning electron microscopy, transmission electron microscopy, X-ray diffractometer, and X-ray photoelectron spectroscopy. TCNQ@$CoWO_4$/CPE was fabricated and used for electrochemical sensing of dopamine. The modified electrode was characterized by cyclic voltammetry, differential pulse voltammetry, amperometry, and electrochemical impedance spectroscopy. The amperometric analysis resulted in a broader linear range of 2 μM to 80

μM for dopamine monitoring and a high sensitivity of 0.202 μA mM^{-1}. EIS analysis provided the R_{ct} value of 950 Ω cm^2. Real sample analysis was performed in commercially available dopamine hydrochloride injections by using the standard addition method and the average recovery result was found to be 95.60 to 99.36%.

Chapter 5 In this chapter, we have synthesized Co@NC and Pd-Co@NC. The synthesized nanoparticles were characterized by X-ray diffractometer, scanning electron microscopy, transmission electron microscopy, and X-ray photoelectron spectroscopy. By using TCNQ and Pd-Co@NC design of a low voltage TCNQ-Pd-Co@NC modified electrode and used for electrochemical sensing of NADH. The modified electrode shows good sensitivity (21.5 μA mM^{-1}), and a low detection limit (5.17 μM). Real sample analysis was performed in the avocado juice sample by using the standard addition method and the recovery result was found to be 95.20 to 99.36%.

6.2 Future Work

In this thesis, we have synthesized transition metal nanoparticles and mixed them with TCNQ to improve the redox behavior and it has been found that the redox properties and stability of TCNQ are increased. We have fabricated modified electrodes for the electrochemical sensing of biomolecules such as ascorbic acid, dopamine, and NADH and performed the real sample analysis in commercially available materials. In the present time, diabetes, depression, cancer, heart and kidney diseases greatly threaten the average person's life so a requirement of a point-care device has been essentially realized. We have proven efficient and stable electrochemical sensing of dopamine, NADH, and ascorbic acid. The methodology can be patented. The methodology, experimental results,

and techniques can be shared with industries involved in medical diagnostics via the transfer of technology and can be commercialized.

- We would develop modified electrode material on screen printing electrodes by spray coating or inkjet printing as a portable sensor device for the detection of ascorbic acid by a non-invasive method. This device can be used for point-of-care (POC) study.
- We can also make efforts to explore the potential of TCNQ-Pd-Co@NC nanocomposite for designing trace-level sensors for environmental pollutants since the atmosphere we breathe, and the water resources we depend on for daily needs are surrounded by a growing number of hazardous pollutants in our environment.