

Synthesis of Co-Pd-Si nanocomposite and its Application in Oxygen Evolution Reaction



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Award of Degree*

DOCTOR OF PHILOSOPHY

By

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Summary and Future scope

5.1 Summary

The results of the thesis are summarized in this chapter, which ends with a suggestion or outlook for further research in this field in the future.

The thesis entitled “Functional nanomaterial-based composites and their biomedical applications” has been divided into five chapters.

1. Introduction
2. Materials and Methods
3. Synthesis and Properties of Organotrialkoxysilane Functionalized Palladium-Cobalt Heterogeneous Catalysts for Oxygen Evolution Reaction
4. Studies on porous nanostructured Palladium-Cobalt-Silica as Heterogeneous Catalysts for Oxygen Evolution Reaction
5. Summary and Future scope

The important outcomes from the present study are as follows:

- ❖ The quest for sustainable and renewable energy solutions has become a pivotal focus in modern scientific research.
- ❖ Among the various strategies explored, the oxygen evolution reaction (OER) stands out as a critical process in technologies such as water splitting for hydrogen production, metal-air batteries, and regenerative fuel cells.

- ❖ The efficiency of these technologies relies heavily on the development of effective and durable catalysts that can facilitate the OER with minimal energy input.
- ❖ Despite the widespread use of noble metal nanocatalysts in heterogeneous catalysis, their catalytic efficiency per noble metal atom has not yet reached the desired levels.
- ❖ This shortfall highlights the need for innovative approaches in catalyst design and synthesis.
- ❖ It also helps in densification of implant material, hence improve the mechanical properties.

Chapter 1: This chapter begins with a quick overview of the history of nanotechnology and then moves on to discuss metal and palladium nanoparticle introductions. The process of electrochemical water splitting, which uses the anodic oxygen evolution reaction (OER) and the cathodic hydrogen evolution reaction (HER) to produce significant volumes of O₂ gas, is economical, efficient, and ecologically benign. The exact catalyst and reaction process that maximises OER's stability and activity are yet unknown, despite its promise. OER thus confronts a great deal of unanswered questions and formidable obstacles. To hasten the commercialization of water electrolyzers, a highly active, dependable, and reasonably priced electrocatalyst must be developed immediately. Since overpotential is crucial in regulating the reaction on metal catalysts and oxygen's capacity to attach to the catalyst surface, the OER mechanism on metal oxides has traditionally been deduced from that on metal catalysts. Ultimately, the current thesis work's history, inspiration, and schedule are also covered.

Chapter 2: Explain in detail the materials and experimental procedures that were utilized to create a variety of materials, such as PdNPs, Co-NTA, Co@NC, Co@PdNPs1,

Co@PdNPs2, Co@PdNPs3, PdNPs1, and PdNPs2. X-ray Diffractometer (XRD), Scanning Electron Microscope (SEM), Transmission Electron Microscopy (TEM), Energy Dispersive Spectroscopy (EDX), BET, and X-ray Photoelectron Spectroscopy (XPS) are used to characterize nanomaterials and nanocomposites in detail. These methods offer in-depth understandings of the materials' surface, compositional, morphological, and structural characteristics. For electrochemical characterisation, cyclic voltammetry (CV) is utilised, whilst electrochemical impedance spectroscopy (EIS) and linear sweep voltammetry (LSV) are applied for electrochemical applications. A brief discussion of each approach is given to give necessary background knowledge.

Chapter 3: Explain the chemical reduction process used to produce Pd-doped bimetallic nanoparticles, or Co@Pdnp, a seed-mediated growth event. The synthetic material was examined using EDX, XRD, TEM, SEM, and XPS. To assess the effects of palladium nanogeometry and nanostructured silica on oxygen evolution reaction (OER), three systems of bimetallic nanocatalysts with varying post-calcination compositions were developed: The following Co@Pdnp values are reported: Si = 4.54%, Pd = 4.36%, Co = 91.10%; Si = 2.81%, Pd = 5.83%, Co = 91.36%; Si = 0.00%, Pd = 9.48%, Co = 90.52%; and Co@Pdnp3: Si = 0.00%. When nanostructured silica is present, it helps to: (a) make the nanocatalyst more recyclable; (b) greatly enhance the palladium nanogeometry; and (c) encourage efficient interaction between the cobalt and palladium components during OER. A nanostructured silica-derived thin film composed of Co@Pdnp produced a very high current density at a low overpotential, with a minor Tafel slope of 39 mV/dec and a catalyst loading of 3.5 mg/cm² on carbon cloth. In the absence of silica, the nanocatalysts were relatively larger and exhibited a lower current density (19 mA/cm²) compared to the higher current density (20.5 mA/cm²) recorded with high silica content.

Chapter 4: This chapter examines how 3-aminopropyltrimethoxysilane (3-APTMS) actively participates in the production of Co@NC, CoPd@NC-1, and CoPd@NC-2, enabling the incorporation of Co-NTA nanowires as precursors. Porous CoPd@NC was developed with different proportions of nanostructured silica in an N-doped carbon matrix in order to control the presence of nanostructured silica following calcination at 700°C, which is advantageous for the oxygen evolution reaction (OER). XRD, TEM, SEM, and EDX were used to analyse CoPd@NC-1, which has a high silica concentration, and CoPd@NC-2, which has a comparatively lower silica content. In comparison to catalysts without nanostructured silica, the addition of nanostructured silica allowed for the development of stabilised bimetallic nanogeometries of cobalt and palladium, which enhanced OER performance. At a catalyst loading of 3.5 mg/cm² on carbon cloth, CoPd@NC-1 and CoPd@NC-2 both produced very high current densities at low overpotentials, with CoPd@NC-1 hitting 0.79 V versus RHE at 10 mA/cm². Both compounds had modest Tafel slopes of 28 mV/dec and 44 mV/dec. When silica was missing, the nanocatalysts were bigger and had lower current densities than when silica was present in greater amounts.

Chapter 5: The results of this study offer important insights into creating advanced nanocatalysts for OER, potentially impacting the wider renewable energy technology field. This research enhances our knowledge of nanoscale catalyst behavior and paves the way for developing more efficient, cost-effective, and durable catalysts for sustainable energy uses. The study employs different characterization techniques to analyze the physicochemical properties of the nanoparticles.

5.2 Future Scope

With the least amount of constraint possible, the thesis study attempts to discover and analyse electrochemical applications based on N doped carbon composites in the scientific community. Even though this covers potential candidates and efficient replacements for the noble metals in the electrochemical application field, there are still a number of significant areas that remain unexplored in other major application areas.

These are as follows.

- The structure and characteristics of the carbon-based polymeric composite may be modified by adding various fillers or nanomaterials. This increases the likelihood of working towards enhancing the polymer's conductivity, mass transfer, and activity in order to introduce it into industrial electrochemical and electronic applications.
- For redox processes using carbon-based polymer composites, a precise and thorough reinvestigation of the reaction mechanism is required since the electrocatalytic process occurs on complex composite structures.
- In order to continually supply and meet energy demands, renewable energy sources must be combined with electrochemical conversion and energy storage.
- To satisfy the demands of industrial applications with performance as high as noble metals, research efforts should be focused on conjugated polymer composites to enhance the redox processes.