




REGULAR ARTICLE

Development of a Promising Zeolitic Imidazolate Framework-Based Nanostructure Cathode Catalyst for Low-Temperature Fuel Cells

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Recently, significant progress has been made in developing alternative catalysts, particularly using metal-organic frameworks (MOFs) like ZIF (zeolitic imidazolate framework)-based materials. ZIF-based catalysts have shown promise in enhancing ORR activity due to their tunable structure, high surface area, and stability at low temperatures. These catalysts provide an effective solution to reducing platinum usage while maintaining or improving the efficiency of the ORR, making them a key component in developing more sustainable and cost-effective fuel cells. The application of ZIF-based cathode catalysts in low-temperature PEMFCs represents a critical advancement in the pursuit of green energy solutions, offering an avenue for improving performance while addressing cost and environmental concerns. In this study, the development of ZIF-based electrocatalyst for ORR in PEMFC applications have been reported, a ZIF-based electrocatalyst such as ZIF-8 is prepared under solvothermal condition using zinc sources (such as $\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$) and 2-methylimidazole (2MeIm). The prepared electrocatalyst is characterized by different surface characterization techniques which is co-related with electrochemical studies. Cyclic voltammetry was done to obtain onset potential of the prepared electrocatalyst at an optimum scan rate in basic medium. Chronoamperometry was done to study the electro-stability of the material.

Keywords: Low temperature fuel cell, Electrocatalyst, Oxygen reduction reaction, Zeolitic imidazolate framework, Sustainable alternative source of energy.

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1. INTRODUCTION

Fuel cells offer a promising alternative to fossil fuels; however, their widespread adoption is hindered by the slow kinetics of the oxygen reduction reaction (ORR) at the cathode and the high cost of platinum-based catalysts. To address these challenges, research has focused on developing platinum-group metal-free (PGM-free) electrocatalysts that can improve ORR efficiency and reduce costs, which is crucial for advancing fuel cell technology towards a more sustainable and economically viable energy solution [1, 2]. Among the potential candidates, Zeolitic Imidazolate Framework (ZIF), a subclass of metal-organic frameworks (MOFs), has emerged as a promising PGM-free catalyst for ORR. The novelty of using ZIF-8 as an ORR catalyst in PEM fuel cells stems from its unique structure, microporosity, high chemical and thermal stability, and large surface area. Additionally, its tunable structure – determined by the choice of metal cations, imidazole linkers, and solvents – provides opportunities for further optimization of catalytic performance [3-13]. Recent research has explored ZIFs as electrocatalysts for ORR due to these

exceptional properties. This study uses a solvothermal method to synthesize ZIF-8 and evaluates its structural, morphological, surface, and electrochemical properties. The goal is to develop an efficient, cost-effective PGM-free electrocatalyst for ORR, advancing fuel cell technology. The synthesized cathode based electrocatalyst was subjected to X-Ray Diffraction (XRD) Analysis, Fourier Transform Infrared Spectroscopy (FTIR), Cyclic Voltammetry (CV), and Chronoamperometry (CA) to investigate its structural and electrochemical properties.

2. MATERIALS AND METHODS

2.1 Materials

The chemicals and reagents used as received in this work without any further purification. For the preparation of ZIF, Zinc acetate dihydrate, 2-methylimidazole (2-MeIm), ethanol, methanol, MWCNTs, sodium dodecylsulfate (SDS), and deionized water were required.

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2.2 Synthesis of ZIF-8

A typical procedure of steam-assisted conversion method for ZIF-8 preparation was implemented. Firstly, Zn (OAc)₂·2H₂O (0.11 g) and 2-methylimidazole (0.41g) were placed in a small Teflon lined stainless steel autoclave which was supported by a Teflon holder. Then 2.0 mL of H₂O was added to the autoclave. For 24 hours, the autoclave containing the sample was placed inside an oven at 120 degrees Celsius for the crystallization to take place. The autoclave was cooled at room temperature, filtered to obtain the solid products, and washed with distilled water, dried, and collected as shown in Fig. 1 [14].

Synthesis of ZIF-8

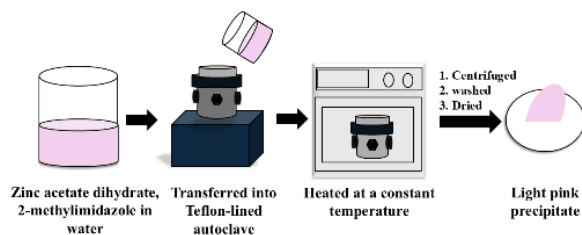


Fig. 1 – Schematic Representation for the synthesis of ZIF-8

2.3 Physical Characterization of the Synthesized Sample

The X-ray diffraction (XRD) analysis was performed using a BRUKER AXS D8 instrument, employing CuK α radiation ($\lambda = 1.5405 \text{ \AA}$). For the XRD measurements, 249.9 mg of ZIF-8 was placed in the sample holder, and a beam of X-rays was directed through the sample using an electron gun. Fourier-transform infrared (FTIR) spectroscopy was conducted with a PerkinElmer FT-IR/FIR spectrophotometer. In the preparation of FTIR pellets, potassium bromide (KBr), a transparent powdered medium, was used as it allows infrared radiation to pass through while absorbing radiation from the sample. The sample and KBr mixture were pressed under high pressure in a pelletizer to form a solid matrix. The KBr facilitated the binding of the sample, enabling the collection of the FTIR spectrum for the synthesized ZIF material.

2.4 Electrode Preparation and Electrochemical Measurements

The three electrodes were assembled which includes the glassy carbon electrode (0.0707 cm²) as the working electrode, the Pt-wire as the counter electrode and the Ag/AgCl electrode as the reference electrode. Prior to the installation of the electrochemical cell, the electrodes were gently rinsed using distilled water and carefully wiped. A small amount (approximately 14 mg) of the prepared ZIF was taken in a centrifuge tube and an adequate amount of ethanol (40 μL) was added to it. Then the mixture was sonicated for few minutes by dipping the centrifuge tube inside the sonicator for its activation. After this, a T-200 micropipette was used to take a

measured volume of the sample (5 μL). Using the micropipette, the ZIF slurry was uniformly dropped cast onto the well-cleaned surface of the glassy carbon electrode (GCE, working electrode) maintaining a constant catalyst loading of 1.75 mg/cm². Then the working electrode was allowed to dry at room temperature. Once the sample dried up, another layer was applied on the electrode (if needed). The three electrodes assembly instrument was used along with EmStats4+ (PalmSens BV Netherlands) and PSTrace 5.9. software. The electrodes were held using crocodile clips and the system was started. The above-mentioned steps were repeated for cyclic voltametric and chronoamperometric studies.

3. RESULTS AND DISCUSSION

3.1 XRD Analysis

X-ray diffraction (XRD) analysis was carried out using an X-ray diffractometer to assess the crystalline characteristics of the catalyst. The XRD pattern of ZIF-8 confirmed its crystalline structure and provided insights into its crystallographic configuration. A significant diffraction peak was observed at a 2θ value of 12.78353°, which was used to calculate the crystallite size of the material. Based on the XRD data (shown in Fig. 2), the crystallite size of the synthesized ZIF-8 was found to be in the nanometer scale, as determined using the Debye-Scherrer equation.

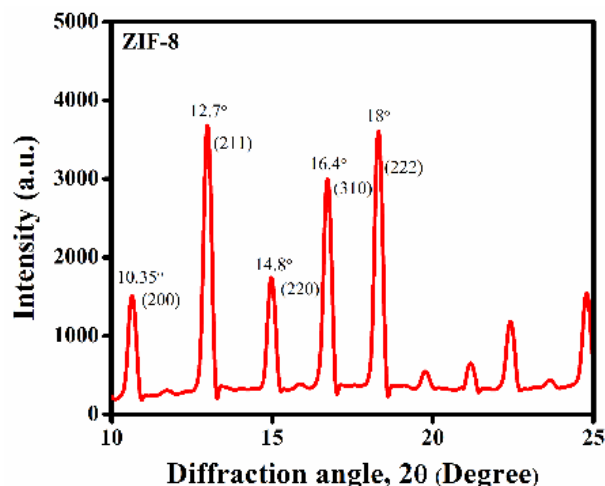


Fig. 2 – X-ray diffraction pattern of the synthesized ZIF-8

3.2 FTIR Analysis

The FTIR spectrum of the ZIF-8 based electrocatalyst reveals characteristic absorption bands that provide insights into its structural and chemical features as shown in Fig. 3. A prominent band typically appears around 1580 cm⁻¹, corresponding to the C = N stretching vibration of the imidazolate linkers in the ZIF-8 framework. This confirms the presence of the metal-organic coordination structure. Additionally, bands in the

range of $400 - 800 \text{ cm}^{-1}$ are associated with the metal-ligand vibrations, particularly the Zn-N stretching, further validating the formation of the ZIF-8 structure. The C-N stretching vibration showed up at the range of 1000 to 1350 cm^{-1} . The peak observed around 1100 cm^{-1} may correspond to the C-C stretching vibrations within the imidazolate rings. The absence or reduction of any significant broadening in the region of bands around $3200 - 3400 \text{ cm}^{-1}$ could indicate minimal surface hydroxylation, implying a hydrophobic surface, favorable for catalysis in the oxygen reduction reaction (ORR). Therefore, the FTIR analysis confirms the integrity of the ZIF-8 framework, with clear evidence of the imidazolate linkers and metal coordination, supporting its potential as an efficient electrocatalyst for ORR. The absence of significant contaminants or decomposition products further ensures the stability of the material under electrochemical conditions.

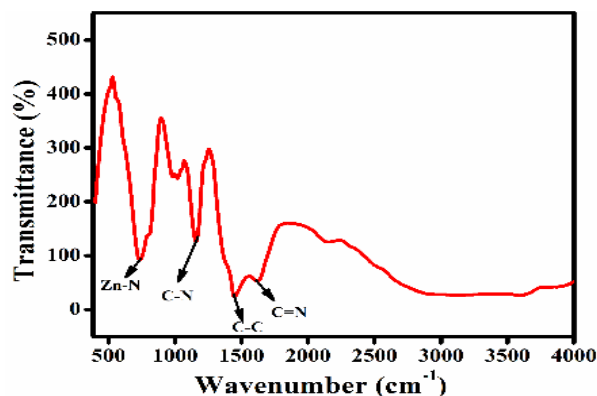


Fig. 3 – FTIR Spectrum of ZIF-8

3.3 Cyclic Voltammetry

The synthesized ZIF-8 is used as a thin film on the glassy carbon electrode to investigate its catalytic behavior towards oxygen reduction reaction (ORR). The electrocatalytic activities of the prepared ZIF are determined by cyclic voltammetry (CV) tests. The measurements are conducted in 0.5 (M) KOH electrolyte. A scan rate variation is executed for the CV wherein six different scan rates i.e., 0.2 V/s , 0.1 V/s , 0.08 V/s , 0.06 V/s and 0.04 V/s are considered. A potential range from -1.5 V to 1.5 V is chosen. Fig. 4 is demonstrating the recorded cyclic voltammogram obtained for ZIF-8 at variable scan rates.

Further for comparison, several electrochemical parameters like onset potential (E_{onset}), peak potential (E_p), peak current densities (i_p) in the forward scans are extracted from CV (as shown in Table 1 and 2) for the prepared ZIF. It can be observed that with the increase in both scan rate and peak potential, the peak current density got enhanced in ZIF-8 (See Table 2). The characteristic single oxidative peak reflects the involvement of the direct conversion of O_2 to H_2O that followed a single pathway mechanism utilizing the prepared cathode based electrocatalyst. Hence the adsorption of an alkaline electrolyte has fulfilled the

desired target of making the reaction to follow a single pathway mechanism that can reduce the complexity of a complex or multi-step reaction (see Fig. 4). In this case, the non-Faradaic regions is almost narrower demonstrating higher performance in the Faradaic zone which starts around at 0.5 V as given in Fig. 4. Additionally, the peak current density is determined for each scan rate and plotted as a function of the scan rate as shown in Fig. 5. The resulting plot indicates that diffusion and adsorption processes are occurring equally. Using McCorry's theory, the electrochemical double layer capacitance (EDLC) values for the prepared ZIF is estimated from the capacitive current as a function of scan rate which further helped in understanding the electrochemical surface area of the solid-liquid interface involved. The tabulated capacitance value at double layer region (C_{dl}) for ZIF-8 is the lowest at the 0.2 V/s with the lowest onset potential of 0.47 V , which is also lower than that of Pt/C (E_{onset} of 0.990 V) [15].

This suggests favorable electrocatalytic performance at the optimal scan rate, characterized by a reduced onset potential. Consequently, ZIF-8 demonstrates potential as a cathode catalyst for the oxygen reduction reaction (ORR) in fuel cells, offering enhanced current density at the optimal scan rate and lowering onset potential.

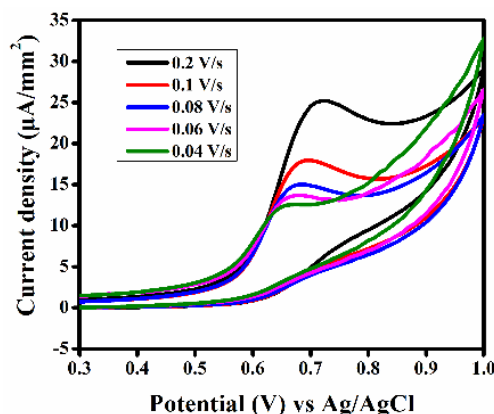


Fig. 4 – Cyclic voltammogram of ZIF-8 at various scan rates

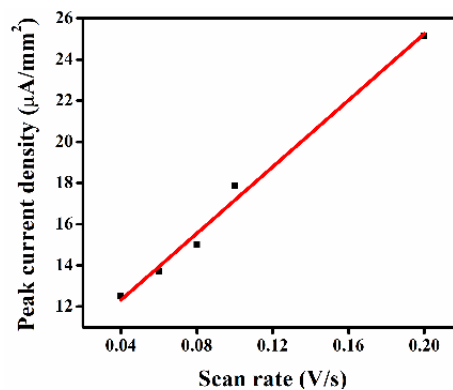


Fig. 5 – Scan rate (V/s) versus Peak Current Density ($\mu\text{A/mm}^2$) for ZIF-8

Table 1 – The Eonset, and their corresponding current density for ZIF-8 at various scan rates are shown below

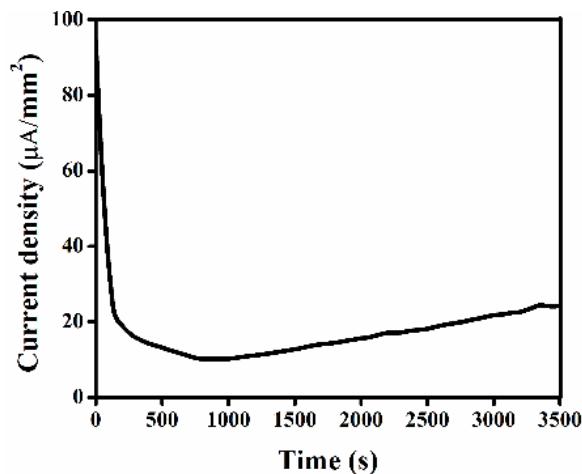
| S. No. | Scan rate (V/s) | Eonset (V) | Current Density ($\mu\text{A}/\text{mm}^2$) at the Corresponding Eonset |
|--------|-----------------|-------------|---|
| 1 | 0.2 | 0.474616752 | 1.95096968 |
| 2 | 0.1 | 0.494612308 | 1.87995083 |
| 3 | 0.08 | 0.499611198 | 2.03974324 |
| 4 | 0.06 | 0.534603421 | 3.56255122 |
| 5 | 0.04 | 0.5446012 | 4.23859601 |

Table 2 – The peak potential, peak current density and EDLC values at various scan rates are shown below

| S. No. | Scan rate (V/s) | Peak Potential (V) | Peak Current Density ($\mu\text{A}/\text{mm}^2$) at the Corresponding Peak Potential | Electrochemically Layer Capacitance (μF) | Double (EDLC) |
|--------|-----------------|--------------------|--|---|---------------|
| 1 | 0.2 | 0.724005776 | 1.95096968 | 40.0895 | |
| 2 | 0.1 | 0.699011331 | 1.87995083 | 60.702 | |
| 3 | 0.08 | 0.684014663 | 2.03974324 | 71.3256 | |
| 4 | 0.06 | 0.679015774 | 3.56255122 | 112.6525 | |
| 5 | 0.04 | 0.664019107 | 4.23859601 | 190.9763 | |

3.4 Chronoamperometry

The chronoamperometry for the synthesized ZIF-8 is recorded to analyze the current retention capacity of the synthesized electro-catalyst within the applied time scale. For this study, the onset potential was obtained from the previously conducted cyclic voltammetry study. The electrochemical stability of ZIF was evaluated by chronoamperometry at 0.6 V in 0.5 (M) KOH electrolyte for which the chronoamperogram for ZIF is recorded for 3600 seconds (about 2 hours) to assess the stability of the electrocatalyst. The chronoamperogram obtained for the synthesized electrocatalyst is given in Fig. 6 which shows that initially there is a drop in the current density. ZIF-8 exhibited the retention of maximum current density and demonstrated its superior performance over the entire time scale. Hence, indicating a superior electrochemical stability for longer period.

**Fig. 6** – Chronoamperogram of ZIF-8

4. CONCLUSIONS

In conclusion, this study demonstrates the successful synthesis of Zeolitic Imidazolate Framework-8 (ZIF-8)-based electrocatalysts through a solvothermal method, which exhibits exceptional performance in catalyzing the oxygen reduction reaction (ORR) within fuel cells. The results confirm that the ZIF-8 electrocatalyst is highly efficient in alkaline environments and meets the criteria for a promising platinum-group-metal-free (PGM-free) alternative. Comprehensive physical and electrochemical characterizations provided valuable insights into the material's composition, morphology, stability, and catalytic activity. X-ray diffraction (XRD) analysis revealed the crystallite size, while Fourier-transform infrared (FT-IR) spectroscopy confirmed the presence of the expected functional groups in the electrocatalyst. Electrochemical testing indicated an optimal scan rate, reduced onset potential, and high current density, with ZIF-8 achieving an impressive onset potential of 0.47 V in an alkaline electrolyte. Overall, these findings highlight the potential of ZIF-8 as a high-performance electrocatalyst for ORR, with versatility for various applications. The novel synthesis approach presented herein offers a valuable strategy for developing efficient cathode-based electrocatalysts, with the potential for further optimization to meet diverse practical requirements.

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Розробка перспективного катодного каталізатора на основі цеолітного імідазолатного каркасу з наноструктурою для низькотемпературних паливних елементів

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Останнім часом досягнуто значного прогресу в розробці альтернативних каталізаторів, зокрема з використанням металоорганічних каркасів (MOF), таких як матеріали на основі ZIF (цеолітового імідазолатного каркасу). Каталізатори на основі ZIF продемонстрували потенціал для підвищення активності ORR завдяки своїй регульованій структурі, великій площі поверхні та стабільності за низьких температур. Ці каталізатори забезпечують ефективне рішення для зменшення використання платини, зберігаючи або підвищуючи ефективність ORR, що робить їх ключовим компонентом у розробці більш стійких та економічно ефективних паливних елементів. Застосування катодних каталізаторів на основі ZIF у низькотемпературних PEMFC є критичним кроком у пошуку рішень для зеленої енергетики, пропонуючи шлях для покращення продуктивності, одночасно вирішуючи проблеми вартості та навколишнього середовища. У цьому дослідженні повідомляється про розробку електрокаталізатора на основі ZIF для ORR у застосуваннях PEMFC, електрокаталізатор на основі ZIF, такий як ZIF-8, готується в сольвотермічних умовах з використанням джерел цинку (таких як $\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$) та 2-метилімідазолу (2MeIm). Підготовлений електрокаталізатор характеризується різними методами характеристики поверхні, що пов'язано з електрохімічними дослідженнями. Циклічну вольтамперометрію було проведено для отримання початкового потенціалу підготовленого електрокаталізатора при оптимальній швидкості сканування в лужному середовищі. Хроноамперометрію було проведено для вивчення електростабільності матеріалу.

Ключові слова: Низькотемпературний паливний елемент, Електрокаталізатор, Реакція відновлення кисню, Цеолітний імідазолатний каркас, Стале альтернативне джерело енергії.