

Synthesis and electrochemical characterization of Pt/C catalysts via polyol method for PEMFC applications

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ABSTRACT

Proton exchange membrane fuel cells (PEMFCs) are promising for clean energy. Here, Pt/C catalysts on Vulcan XC-72 were synthesized via a polyol method using ethylene glycol and polyvinylpyrrolidone. XRD confirmed face-centered cubic Pt with ~1.8 nm crystallites. BET analysis showed a mesoporous structure with a surface area of 91.97 m²/g. SEM-EDX revealed uniform Pt dispersion with 30.18 wt% loading. Electrochemical impedance spectroscopy indicated low solution resistance and moderate charge transfer resistance (~260 Ω), suggesting charge-transfer-controlled kinetics. Linear sweep voltammetry demonstrated good ORR activity, with a current density of -7.2 mA/cm² at -0.8 V. The catalyst showed acceptable stability after 100 cycles. Overall, the synthesized Pt/C catalyst exhibited preliminary ORR activity in alkaline electrolyte, although additional electrochemical evaluation in acidic media together with long-term durability testing is required before practical PEMFC applicability can be confirmed.

Keywords: Pt/C catalyst; Polyol synthesis method; Oxygen reduction reaction (ORR); Proton exchange membrane fuel cells (PEMFCs); Electrochemical characterization.

1. INTRODUCTION

In the context of increasingly severe climate change and environmental pollution, the demand for clean and sustainable energy sources has become a global priority [1, 2]. Excessive reliance on fossil fuels not only depletes natural resources but also significantly increases greenhouse gas emissions, including CO₂ and NO_x, thereby accelerating the greenhouse effect and contributing to extreme weather events [1, 3]. Consequently, the development of environmentally friendly alternative energy technologies is essential for sustainable development and carbon emission reduction. Among the various clean energy technologies, proton exchange membrane fuel cells (PEMFCs) are regarded as one of the most promising solutions because they directly convert chemical energy into electrical energy with high efficiency and near-zero emissions [4-6]. PEMFCs operate at relatively low temperatures, exhibit rapid start-up, and possess a compact design, making them well suited for transportation and portable applications [4, 5]. However, the performance and durability of PEMFCs are highly dependent on the quality of electrocatalysts, particularly for the oxygen reduction reaction (ORR) at the cathode.

Presently, platinum (Pt) is regarded as the most effective electrocatalyst for PEMFCs owing to its high activity and stability [7, 8]. Nevertheless, its high cost and limited availability remain major barriers to large-scale commercialization [8, 9]. To address this issue, considerable efforts have focused on reducing Pt loading by dispersing Pt nanoparticles onto conductive carbon supports to form platinum/carbon (Pt/C) catalysts [10-12]. Pt/C catalysts increase the electrochemically active surface area, improve charge transfer, and reduce catalyst cost. However, challenges such as nanoparticle agglomeration, catalyst degradation, and carbon support corrosion under electrochemical conditions still remain [13-15].

In recent years, the polyol method has attracted considerable attention as a versatile approach for synthesizing Pt/C electrocatalysts because it enables simultaneous reduction of platinum precursors and controlled nanoparticle growth under relatively mild conditions [16, 17]. Recent studies have demonstrated that microwave-assisted and conventional polyol routes can produce Pt/C catalysts with Pt crystallite sizes of only a few nanometers and favorable ORR activity, although catalyst performance remains highly dependent on synthesis temperature, stabilizing agents, carbon support characteristics, and post-treatment conditions [16, 18]. Consequently, establishing reliable correlations between synthesis conditions, structural characteristics, and electrochemical behavior remains an important topic.

Although polyol-derived Pt/C catalysts have been extensively investigated, previous studies have primarily focused on catalyst preparation or catalytic performance alone [16-18]. Comparatively fewer studies have systematically correlated crystallographic structure, morphology, surface properties, impedance behavior, and electrochemical characteristics obtained from complementary characterization techniques. In the present work, Pt/C catalysts supported on Vulcan XC-72 were synthesized via a simple EG/PVP-assisted polyol route. Rather than proposing a new catalyst composition through alloying or support modification, this work aims to establish the relationship between structural characteristics and preliminary electrochemical behavior of a baseline Pt/C catalyst prepared by a straightforward polyol method. The obtained results provide useful reference data for subsequent optimization of Pt-based electrocatalysts for PEMFC-related applications.

2. EXPERIMENTAL SECTION

2.1. Materials

Ethylene glycol (EG, Merck), hexachloroplatinic acid (H_2PtCl_6 , Sigma-Aldrich), polyvinylpyrrolidone (PVP, Merck), carbon black Vulcan XC-72 (Cabot Corporation), and Nafion solution (5 wt%, DuPont) were used as received without further purification. Nitric acid (HNO_3 , 0.1 M) and sulfuric acid (H_2SO_4 , 0.5 M) were used for electrochemical treatment. Deionized water was used throughout the experiments.

2.2. Experiment preparation

2.2.1. Synthesis of Pt/C catalysts

Pt/C catalysts were synthesized via a polyol method using ethylene glycol (EG) as both solvent and reducing agent. In a typical procedure, 45 mL of EG was heated to 150 °C under continuous stirring. Subsequently, 0.25 g of polyvinylpyrrolidone (PVP) was added and the solution was stirred for 30 min to ensure complete dissolution and stabilization of the system. Afterward, 0.25 g of $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$ was introduced into the reaction mixture, and the temperature was maintained at 150 °C for 10 - 15 min to allow the reduction of Pt^{4+} to metallic Pt nanoparticles. Then, 0.2 g of Vulcan XC-72 carbon was added to the mixture, followed by ultrasonic treatment to achieve uniform dispersion and promote the deposition of Pt nanoparticles onto the carbon support. After completion of the reaction, the mixture was naturally cooled to room temperature. The obtained Pt/C product was collected by centrifugation, washed several times with deionized water and ethanol to remove residual impurities, and finally dried at 70 °C to obtain the final catalyst.

2.2.2. Electrode preparation

The catalyst ink was prepared by dispersing 5 mg of Pt/C in 1 mL of isopropanol, followed by the addition of 10 μL of Nafion solution (5 wt%). The mixture was ultrasonicated for 1-2 h to obtain a homogeneous suspension.

A calculated volume of the catalyst ink was then drop-cast onto a pretreated glassy carbon electrode and dried at 70 °C prior to electrochemical measurements.

2.2.3. Characterization and electrochemical measurements

The crystal structure of the synthesized materials was characterized by X-ray diffraction (XRD,

X'Pert PRO, Malvern Panalytical, Netherlands) using Cu K α radiation ($\lambda = 0.15406$ nm).

The specific surface area was determined by N₂ adsorption-desorption measurements at 77 K using a BET analyzer (ASAP 2020 or TriStar II 3020, Micromeritics, USA). The morphology and microstructure of the samples were examined by scanning electron microscopy (SEM, JEOL JEM-2800), while the elemental composition was analyzed using energy-dispersive X-ray spectroscopy (EDX, JEOL JSM-7610F). Electrochemical measurements were carried out using a conventional three-electrode system, consisting of a glassy carbon electrode loaded with catalyst as the working electrode, an Ag/AgCl electrode as the reference electrode, and a Pt wire as the counter electrode. All potentials were converted to the reversible hydrogen electrode (RHE) scale. Cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) measurements were performed using a Gamry Reference 600+ system in alkaline electrolyte (KOH 1M). CV measurements were conducted in the potential range of 0 - 1.4 V vs RHE at a scan rate of 50 mV/s. EIS measurements were carried out over a frequency range from 100 kHz to 0.1 Hz, and the obtained data were fitted using an equivalent circuit model ($R_s-(R_{ct}||CPE)$). Linear sweep voltammetry (LSV) was performed using an Autolab PGSTAT302N (Metrohm Autolab) to evaluate the oxygen reduction reaction (ORR) activity in alkaline medium.

3. RESULTS AND DISCUSSION

3.1. Structural and morphological characterization of Pt/C catalysts

The X-ray diffraction pattern of the Pt/C catalyst (Figure 1c) exhibits characteristic peaks at approximately $2\theta \approx 40^\circ$, 47° , and 70° , corresponding to the (111), (200), and (220) planes of face-centered cubic (fcc) metallic Pt [16]. The presence of these peaks confirms that Pt exists predominantly in the metallic state, while no characteristic peaks of Pt oxides are observed within the detection limit of the technique. The relatively broad diffraction peaks indicate small crystallite size; the average crystallite size calculated using the Scherrer equation is approximately 1.8 nm, which is typical for nanostructured catalysts.

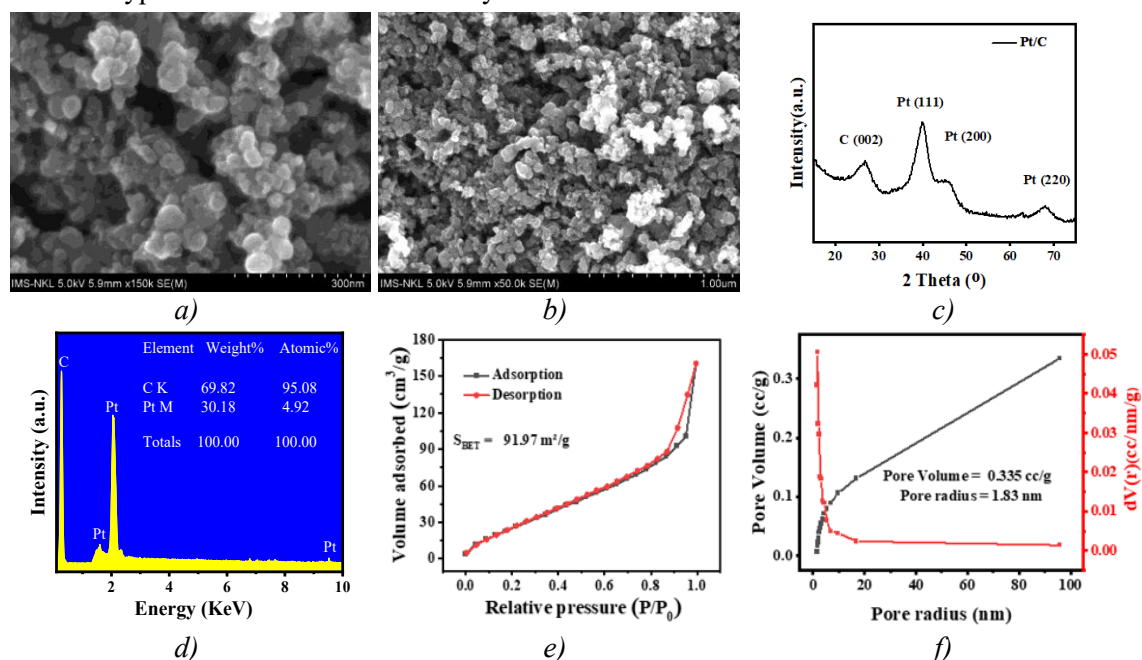


Figure 1. SEM images of the Pt/C catalyst (a, b); X-ray diffraction (XRD) pattern (c); EDX spectrum (d); and N₂ adsorption-desorption isotherms and pore size distribution (BET analysis) (e, f).

SEM images (Figures 1a, b) reveal Pt particles with apparent sizes in the range of 30 - 50 nm, which are relatively uniformly distributed on the surface of Vulcan XC-72 carbon. This distribution suggests that the polyol method effectively controls the formation and anchoring of Pt particles on the carbon support. The average crystallite size (~ 1.8 nm) calculated from the Scherrer equation represents the coherent diffraction domain of individual Pt nanocrystals rather than the physical particle size. By contrast, SEM mainly visualizes secondary particles or aggregates formed by multiple Pt nanocrystals deposited on the rough carbon support. Moreover, the limited spatial resolution of conventional SEM makes individual Pt nanocrystals difficult to distinguish. Therefore, the apparent particle size of 30–50 nm observed in SEM most likely corresponds to agglomerated clusters composed of numerous Pt nanocrystallites. Nevertheless, high-resolution transmission electron microscopy (TEM) would provide more direct evidence of Pt nanoparticle size and dispersion and will be considered in future work.

EDX analysis confirms that the material mainly consists of carbon and platinum, with a Pt content of approximately 30.18 wt%, and no significant impurities are detected. This result indicates that the synthesized material possesses high purity and is suitable for electrochemical catalytic applications.

BET analysis shows that the material has a specific surface area of $91.97 \text{ m}^2/\text{g}$ and exhibits a mesoporous structure [11, 12, 19].

3.2. Electrochemical properties

3.2.1. Cyclic voltammetry

The cyclic voltammetry (CV) curves of the Pt/C electrode recorded in 1 M KOH are shown in Figure 2a exhibit characteristic electrochemical features of Pt under alkaline conditions [7]. In the low potential region (0-0.35 V vs RHE), weak hydrogen adsorption/desorption features can be observed, which may be attributed to the competitive adsorption of hydroxyl species (OH^-) on the Pt surface, suppressing hydrogen underpotential deposition in alkaline medium.

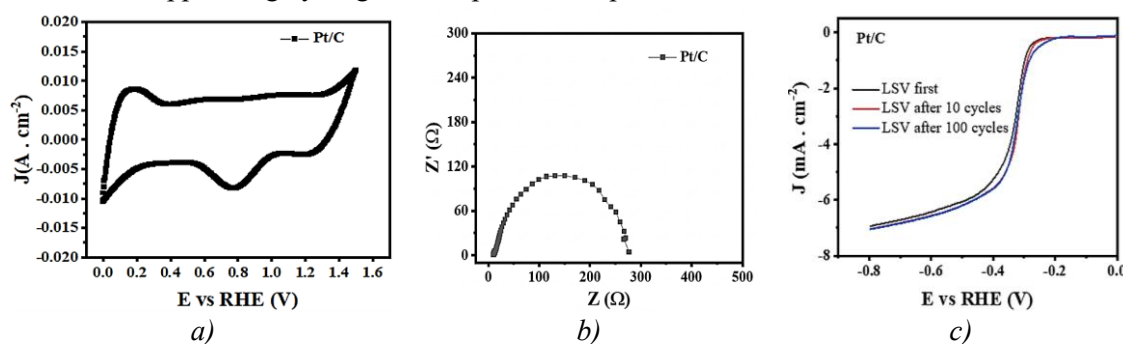


Figure 2. Cyclic voltammetry (CV) curves of the Pt/C catalyst in alkaline electrolyte (a); Nyquist plot of the Pt/C electrode in alkaline electrolyte (b); Linear sweep voltammetry (LSV) curves of the Pt/C catalyst (c).

In the intermediate potential region (~ 0.35 - 0.9 V vs RHE), the current remains relatively stable, indicating predominantly double-layer capacitive behavior with minimal Faradaic contribution. As the potential increases beyond ~ 0.9 - 1.0 V vs RHE, a gradual rise in anodic current is observed, which becomes more pronounced above ~ 1.2 V vs RHE. This behavior is attributed to the formation of Pt oxide/hydroxide species (Pt-OH/Pt-O) on the electrode surface, along with the possible onset of the oxygen evolution reaction at higher potentials.

During the reverse scan, a cathodic peak appears at approximately 0.7 - 0.85 V vs RHE, corresponding to the reduction of the previously formed Pt oxides. The hysteresis between the forward and reverse scans reflects the partially irreversible nature of oxide formation and reduction processes, as well as contributions from capacitive charging.

Although the CV response confirms the presence of electrochemically active Pt surface sites, quantitative ECSA was not calculated in this study because the experimental protocol was not optimized for standardized hydrogen adsorption/desorption or CO-stripping analysis. Further electrochemical measurements under controlled conditions are required for accurate ECSA determination.

3.2.2. Electrochemical impedance spectroscopy

The Nyquist plot of the Pt/C electrode in 1 M KOH (Figure 2b) exhibits a distinct semicircle, indicating that the electrochemical response is mainly governed by the charge transfer process at the electrode-electrolyte interface [13]. The obtained data were fitted using an equivalent circuit model $R_s-(R_{ct}||CPE)$, yielding a solution resistance $R_s \approx 10 \Omega$ and a charge transfer resistance $R_{ct} \approx 260 \Omega$. The low R_s value is consistent with the high ionic conductivity of the alkaline electrolyte and good electrode-electrolyte contact.

The absence of a pronounced Warburg diffusion tail at low frequencies suggests that mass transport limitations are negligible, likely attributed to the mesoporous structure of the Vulcan XC-72 carbon support facilitating efficient ion diffusion to the active Pt sites. The slightly depressed semicircle, characterized by a CPE exponent $n < 1$, further indicates surface heterogeneity arising from the distribution of Pt nanoparticles on the carbon support, which is consistent with the SEM observations.

3.2.3. Linear sweep voltammetry

The oxygen reduction reaction activity of the Pt/C catalyst was evaluated using linear sweep voltammetry (LSV), as shown in Figure 2c. The polarization curve shows a gradual increase in current density with decreasing potential, reaching a limiting current density of approximately -7.2 mA cm^{-2} at -0.8 V vs RHE, indicating promising ORR activity in alkaline medium [6, 10]. Compared with the limiting current density of approximately -5.6 mA cm^{-2} reported for a commercial Pt/C catalyst, the catalyst developed in this work exhibited enhanced electrocatalytic activity, highlighting its potential for further optimization [20]. The relatively favorable onset potential further suggests the presence of active Pt sites contributing to the reaction.

The electrochemical stability of the catalyst was assessed through repeated potential cycling. After 100 cycles in alkaline electrolyte, the LSV curves exhibit only a slight decrease in current density while largely retaining their original shape, indicating preliminary stability under the tested conditions [14, 15]. The present durability evaluation represents only an initial assessment and is insufficient to evaluate long-term degradation phenomena, including Pt dissolution, particle coalescence, Ostwald ripening, and carbon support corrosion. More rigorous accelerated durability tests involving several thousand potential cycles will therefore be performed in future work. The ORR measurements were conducted in alkaline electrolyte to provide a preliminary evaluation of the intrinsic electrocatalytic behavior of the synthesized Pt/C catalyst. Since practical PEMFCs generally operate under acidic conditions, the electrochemical performance obtained in this study should not be directly interpreted as actual PEMFC performance. Future work will therefore include electrochemical evaluation in acidic electrolyte together with membrane-electrode assembly (MEA) testing to verify practical PEMFC performance.

4. CONCLUSIONS

Pt/C catalysts supported on Vulcan XC-72 were synthesized through an EG/PVP-assisted polyol method. Characterization confirmed fcc Pt crystallites of approximately 1.8 nm, a Pt loading of 30.18 wt%, and a mesoporous structure with a specific surface area of $91.97 \text{ m}^2/\text{g}$. In 1 M KOH, the catalyst exhibited an ORR current density of approximately -7.2 mA cm^{-2} and a charge-transfer resistance of $\sim 260 \Omega$. Its electrochemical response was largely retained after 100 cycles, indicating preliminary stability. Nevertheless, evaluation in acidic electrolyte, standardized ORR testing, and long-term accelerated durability tests are required before its applicability to PEMFCs can be established.

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TÓM TẮT**Tổng hợp và đặc trưng điện hóa xúc tác Pt/C bằng phương pháp polyol cho ứng dụng pin nhiên liệu màng trao đổi proton**

Pin nhiên liệu màng trao đổi proton (PEMFC) được xem là một trong những giải pháp năng lượng sạch đầy triển vọng. Trong nghiên cứu này, xúc tác Pt/C trên nền carbon Vulcan XC-72 được tổng hợp bằng phương pháp polyol sử dụng ethylene glycol và polyvinylpyrrolidone. Kết quả nhiễu xạ tia X (XRD) xác nhận sự hình thành pha Pt cấu trúc lập phương tâm mặt với kích thước tinh thể khoảng ~1,8 nm. Phân tích BET cho thấy vật liệu có cấu trúc mao quản trung bình với diện tích bề mặt riêng đạt 91,97 m²/g. Ảnh hiển vi điện tử quét kết hợp phân tích EDX cho thấy Pt phân tán đồng đều với hàm lượng khoảng 30,18% khối lượng. Phổ trở kháng điện hóa (EIS) cho thấy điện trở dung dịch thấp và điện trở chuyển điện tích ở mức trung bình (~260 Ω), gợi ý cơ chế phản ứng bị chi phối bởi quá trình chuyển điện tích. Phép đo voltammetry quét tuyến tính (LSV) cho thấy hoạt tính tốt đối với phản ứng khử oxy (ORR), với mật độ dòng đạt -7,2 mA/cm² tại -0,8 V. Xúc tác cũng thể hiện độ ổn định chấp nhận được sau 100 chu kỳ. Nhìn chung, xúc tác Pt/C tổng hợp được cho thấy hoạt tính ORR bước đầu trong môi trường điện ly kiềm; tuy nhiên, cần tiếp tục đánh giá điện hóa trong môi trường axit và thực hiện các thử nghiệm độ bền dài hạn trước khi có thể khẳng định khả năng ứng dụng thực tế của xúc tác trong pin nhiên liệu màng trao đổi proton.

Từ khoá: Xúc tác Pt/C; Phương pháp tổng hợp polyol; Phản ứng khử oxy (ORR); Pin nhiên liệu màng trao đổi proton (PEMFC); Đặc trưng điện hóa.