

Research Article

Ir Supported by Mesoporous SiO₂ (SBA15) as the Catalyst for Proton Exchange Membrane Water Electrolyzer

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Abstract

The oxygen evolution reaction (OER) is a critical process in proton exchange membrane (PEM) water electrolysis, but its sluggish kinetics and harsh operational environment e.g., high anodic potentials, oxidative atmosphere, and strong acidic media pose significant challenges to catalyst design. Iridium (Ir) is among the most effective catalysts for OER in acidic conditions; however, its scarcity and high cost necessitate strategies to enhance its efficiency and stability. Reducing the size of Ir nanoparticle to 3-4 nm can significantly enhance OER activity due to an increased surface area and more accessible active sites. In this study, we report the synthesis of a highly active and stable Ir/SBA15 catalyst, in which Ir nanoparticles are uniformly deposited onto mesoporous silica (SBA15). The mesoporous structure of SBA-15 facilitates uniform nanoparticle dispersion, while the incorporation of a conductive nanochain structure enhances electronic conductivity. Transmission electron microscopy (TEM) confirmed the uniform distribution of Ir nanoparticles with an average size of 3-4 nm. Electrochemical tests revealed that Ir/SBA15 exhibits high OER activity, excellent cycling stability, and improved conductivity compared to conventional Ir catalysts. These findings demonstrate that SBA15 supported Ir nanochains offer a promising pathway for efficient and durable OER catalysis in PEM electrolyzers, achieving a balance between catalytic performance, structural integrity, and long-term operational stability.

Keywords

Proton Exchange Membrane Water Electrolysis, Iridium, Mesoporous Silica, Oxygen Evolution Reaction

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1. Introduction

Proton-exchange membrane (PEM) water electrolysis is expected to play a key role in hydrogen production for mobility and energy storage, as it leverages its efficiency, ability to quickly cycle up and down, and ability to deliver hydrogen at high and differential pressures [1-4]. It is also expected that the capital cost for commercial electrolyzer will become competitive after combining with lost-cost and renewable sources such as solar and wind [5-8]. However, the challenge in the PEM water electrolysis process is the anodic oxygen evolution reaction (OER) because of its extremely sluggish kinetics compared with the hydrogen evolution reaction (HER) on the cathode side, where a small mass loading of Pt/C catalyst is used as the catalyst [9-11]. Noble metals, such as Iridium (Ir) based metal oxides are the typical OER materials in acidic electrolyzers due to their reasonable activity and durability [12, 13]. Two typical noble platinum and ruthenium metals are another two examples. The first one requires a high overpotential while the latter one has durability concerns [14, 15]. However, the low Earth abundance of Ir metals severely limits the global deployment of PEM water electrolyzers [16]. To balance the limited Earth reserves with the growing demand for PEM electrolyzer deployment, the most effective approach is to enhance the catalytic performance of Ir catalysts, thereby reducing the required mass loading.

Recent efforts to develop enhanced anode catalysts for acidic electrolyzers have mainly focused on Ir oxide supported by chemically stable metal oxides, such as titania, or alloying Ir with nickel and copper or other noble metals (e.g., Pt and Ru) to improve the durability and performance [17-20]. IrO₂ and Ir nanoparticles supported on high surface area material are another effective approach to reduce the amount of noble metal [21]. This strategy not only stabilizes the initial catalyst surface area but also significantly improves the mass-based activity of the catalysts. Carbon black (e.g. XC-72), a typical fuel cell catalyst support material, is not chemically stable in the strong oxidative conditions of water electrolysis, leading to the aggregation and migration of noble metal catalysts and even their detachment from the support surface, resulting in a loss of the electroactive surface area. Therefore, various acid-tolerant metal oxides including modified TiO₂, Nb₂O₅, and antimony doped tin oxide (ATO) have been investigated as an alternative to carbon [22-25]. A recent study reported that TiO₂ supported Ir@IrO(OH)_x core-shell nanoparticles could be efficient, lost-cost and stable catalysts for electrochemical water splitting [23]. Further studies also demonstrated that Nb₂O_{5-x} supported iridium exhibited exceptional performance in scalable water electrolyzers (1.839 V @ 3 A cm⁻², and no activity decay during a 2000 h test at 2 A cm⁻²) [24]. In this paper, iridium nanoparticles were uniformly deposited on SBA-15 (also an acid-tolerant oxide), referred to as Ir/SBA-15, which exhibits enhanced conductivity and sub-

sequently improves OER performance. The TEM and OER test demonstrated that the sample exhibited high OER performance and cycle durability, indicating that this sample could be a promising OER catalyst for PEM electrolyzer.

2. Experimental Section

Mix 1.2 g of sodium hydroxide pellets into 150 ml ethylene glycol to make a 0.2 M NaOH solution with stirring and applying a little heat. Add 0.7 g support SBA15, horn sonicate and stir for 45 minutes. Add 0.7 g metal precursor (IrCl₃.xH₂O) and continue mixing for 3 hours. A homogeneously dispersed caramel yellow liquid mixture was obtained and then was heated to 175 °C and reacted for 3 hours, with a temperature control using a thermometer. The resulting mixture was cooled down, removed from the heat, and mixed into 1.5 L of DI water. While mixing, slowly add nitric acid with a pipette until the solution pH is approximately 1 (with the help of litmus paper). Continue to mix for 3 hours. After precipitation, the bottom particles were washed with DI water through a centrifuge 3 times, until a clear supernatant was obtained. The collected nanoparticles were dried in a vacuum oven at 110 °C for 4 hours, named as Ir/SBA15.

The resulting iridium loading onto the SBA15 support was explored using Energy Dispersive X-ray Spectroscopy (EDS) built inside a Phenom ProX Scanning Electron Microscopy, using an electron accelerating voltage of 15 kV and a working distance of 20 mm. The morphology and crystal structure of iridium particles and its distribution on support were investigated by a transmission electron microscope (TEM) (JEOL, model JEM- 2100) operating at 200 kV and X-ray diffraction (XRD, Rigaku Minix-II with Cu K α (1.5406 Å) radiation, 30 kV/15 mA current), respectively. Electrochemical experiments were carried out in a three-electrode electrochemical cell equipped with a water jacket. A Pt wire and a double-junction Hg/HgSO₄ electrode (Pine Research Instruments) were used as the counter and reference electrodes, respectively. Polarization curves for the oxygen evolution reaction were recorded using a VersaSTAT 3 potentiostat (Princeton Applied Research). The electrolyte used in the electrochemical tests was 0.5 M sulfuric acid (H₂SO₄) solution (Sigma-Aldrich). Membrane electrode assemblies (MEAs) were fabricated using either Ir/SBA15 or commercial Ir black as anode electrocatalysts, and Pt/C (40 wt%) as the cathode catalyst. To formulate catalyst inks, the powders were dispersed in a 1:1 volumetric mixture of isopropyl alcohol and deionized water. Nafion® solution (5 wt%) was subsequently added to achieve an ionomer content of 30 wt% for anode and 40 wt% for cathode inks. The resulting mixtures were ultrasonicated in a chilled water bath for a minimum of one hour to ensure uniform dispersion. The catalyst layers were first sprayed onto PTFE substrates, which were then hot-pressed with the N115 membrane at 135 °C for 3 minutes under 2 tons

of pressure. The PTFE backing was removed post-pressing, yielding catalyst-coated membranes (CCMs) with optimized loadings of 2 mg Ir/cm² for the anode and 1 mg Pt/cm² for the cathode. The resulting CCMs were stored in deionized water prior to testing. Electrolyzer cells were constructed using 1 mm thick titanium felt as the porous transport layer on both electrode sides. Assembly torque was standardized to 5 N m, and the active electrode area was fixed at 4 cm². Performance tests were carried out at 80 °C using distilled water delivered at a rate of 40 mL/min. Current-voltage characteristics were obtained across a current density range of 0.1 to 2 A/cm². Durability was assessed through chronopotentiometric measurements at 2 A/cm² over 1000 hours.

3. Results and Discussion

Ir nanoparticles, ranging from 3 to 5 nm, were uniformly deposited on SBA-15 to form Ir/SBA-15. TEM images of Ir/SBA-15 are shown in Figure 1, where SBA-15 exhibits parallel and uniform channels (Figure 1a). Electrical conductivity measures revealed that the Ir/SBA15 catalyst exhibits electronic conductivity comparable to that of commercially available IrO₂ catalyst, suggesting its potential as a viable alternative for PEM electrolyzer applications.

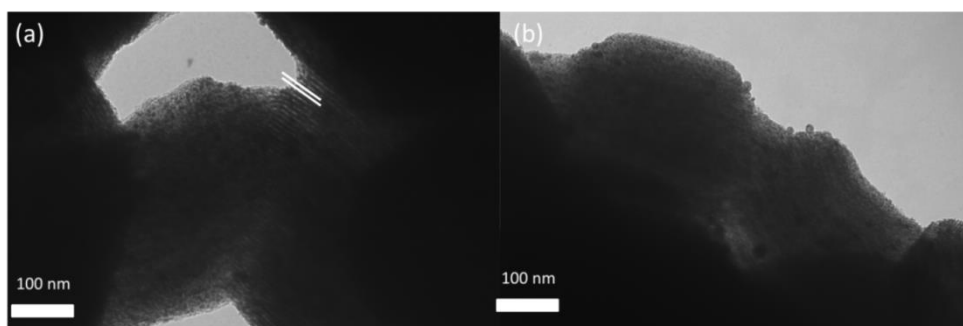


Figure 1. TEM images of Ir/SBA-15.

The XRD pattern (Figure 2a) of Ir/SBA-15 displays a broad diffraction peak centered around $2\theta \approx 22^\circ$, which is characteristic of the amorphous silica framework of SBA-15. In addition, weak diffraction peaks corresponding to metallic Ir are observed, indicating the presence of small, well-dispersed Ir nanoparticles. The X-ray photoelectron spectroscopy (XPS) analysis (Figure 2b) of Ir/SBA-15 revealed two characteristic peaks at binding energies of 64.5 eV and 61.5 eV, which correspond to the Ir 4f 5/2 and Ir 4f 7/2 orbitals, respectively. These values are consistent with

metallic iridium (Ir⁰), indicating that the iridium species exist predominantly in the zero-valent state. Additionally, the structural and morphological characterization using XRD and TEM, along with XPS results, clearly confirmed the successful deposition of Ir nanoparticles on the mesoporous SBA-15 support. The uniform distribution of Ir nanoparticles observed in the TEM images further supports the conclusion that SBA-15 acts as a stable and effective host for well-dispersed Ir species in this composite catalyst system.

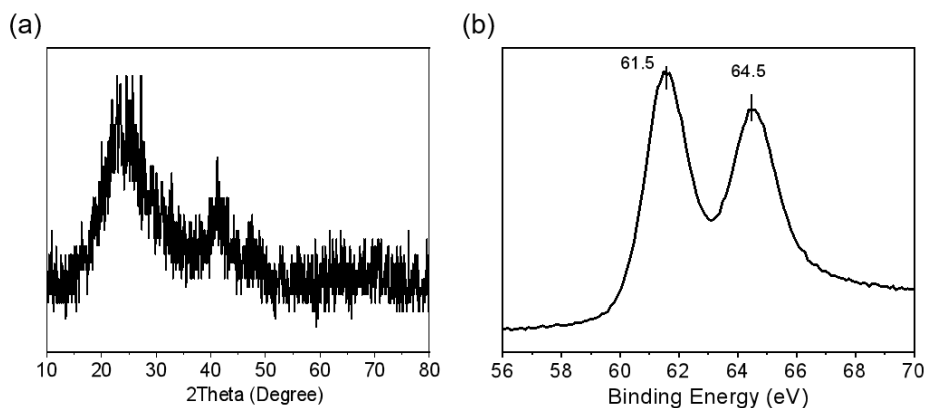


Figure 2. XRD (a) and XPS (b) pattern of Ir/SBA15.

The electrochemical catalytic properties of OER for Ir/SBA15 were examined in 0.1 M HClO₄ acid solution. Figure 3a shows the OER performance of Ir/SBA15 catalyst under the potential window of 1.3 and 1.85 V vs RHE at the scan rate of 5 mv/s. For the comparison, the OER performance for commercial Ir black was also investigated in the same conditions. As can be seen from Figure 3a, Ir/SBA15 exhibited higher OER performance than commercial Ir black. The enhanced OER performance for Ir/SBA15 is ascribed to the fact that smaller nanoparticles can provide more active sites, and the Ir nanoparticles conductive network can significantly enhance the charge transfer for the OER. More interestingly, the anodic and cathodic peaks for Ir/SBA15 are almost overlapping, indicating that rapid electrochemical equilibrium reaction with no faradaic and chemical rate-determining reaction step. The overlapped anodic and cathodic profiles indicate the one-electron electrochemical rate-determining step and the similar valence state between the transition state and the final state of the rate-determining step. The cycle stability of Ir/SBA15 was further examined with 10000 cycles, as shown in Figure 3b. Only limited loss for the OER performance of Ir/SBA15 was observed, and

after 2000 cycles, the OER performance of Ir/SBA15 could be slightly increased, which is attributed to the oxidation state of Ir and more active sites could be provided. Under OER conditions, the Ir nanoparticles could be further oxidized to a higher chemical state and then increase the OER performance. The Tafel slope for Ir/SBA15 and commercial iridium black is 38 and 43 mV/dec, respectively, also indicating that the fast kinetics could be obtained for our Ir/SBA15 catalyst. The cyclic voltammetry (CVs) of Ir/SBA15 in Figure 3c showed the characteristic charging/discharging peaks of redox pseudo-capacitance and double layer capacitance. For the initial CV profile, no significant peaks could be observed, after 2000 cycles under OER conditions, the surface of iridium could be oxidized to high Ir-oxide species, which can be demonstrated by the two new redox peaks (Ir/Ir(OH)₃) between 0.4 and 0.8 V vs RHE and Ir^{III}/Ir^{IV} at the potential of 1~1.4 V vs RHE, respectively). Ir metal nanoparticles was initially oxidized to Ir(OH)₃ in the voltage region between 0.4 and 0.8 V, and further to an hydrous Ir (IV) oxides species beyond the potential of 1.0 V [26]. The cycle test under high OER potential involves the irreversible chemical oxidation of the surface of iridium.

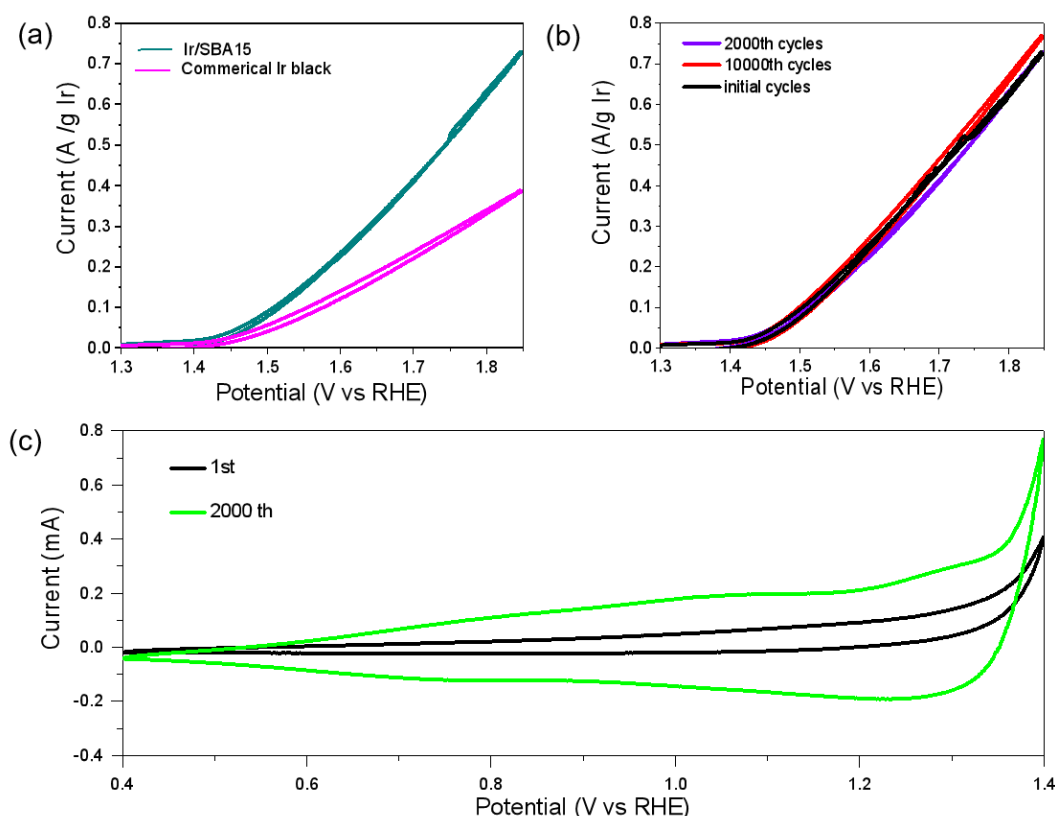


Figure 3. The initial OER performance for Ir/SBA15 and commercial Iridium black (a), cycle durability for Ir/SBA15 (b) and CVs for Ir/SBA15 at potential windows range from 0.4 V to 1.4 V vs RHE (c).

Three-electrode rotating disk electrode (RDE) half-cell tests are an important tool to assess the short-term, small-scale performance of a new electrocatalyst. Years of

catalyst development in the PEM water electrolyzer area have shown, however, that RDE ultimately remains a screening tool that often fails to reliably predict activity or

stability in a full galvanic or electrolytic device. Thus, to assess the performance of our novel Ir/SBA15 catalysts under more practical work conditions, single Membrane Electrode Assemblies (MEAs) were prepared using the Ir/SBA15 as anode, commercial N-115 membranes, and commercial Pt/C catalyst as cathode electrocatalysts. Their water splitting performance was then evaluated in a realistic single polymer electrolyte membrane water electrolysis cell (Figure 4a). Again, for comparison, commercial iridium black was evaluated under the same conditions. The MEA performance of Ir/SBA15 is superior to the samples of standard commercial Ir black, exhibiting the current density of 2 A cm^{-2} at 1.75 V . Water splitting potentials of standard commercial Ir black reached 1.78 V at the same current densities of 2 A cm^{-2} , respectively. The superior electrochemical performance of the electrolyzer using Ir/SBA15 can be further confirmed by the lower high frequency resistance (HFR) at various current

densities (Figure 4b). Therefore, it is understandable that a slight potential decrease could be observed for the Ir/SBA15-based proton exchange membrane electrolyzer. Long-term testing results showed that the voltage decay of the electrolyzer using Ir/SBA15 was around 25 mV after a 1000-hour durability test, compared to approximately 80 mV for the Ir black catalyst under the same testing conditions. Besides, during the first fifty hours, the water splitting potential of Ir/SBA15 slightly decreased compared with that of start-up. It is noted that two observed redox peaks and increased OER performance can be observed (Figure 3c), indicating that more active sites will be provided after the cycle test during OER working potential (around 1.8 V). However, we still observed a slight performance degradation after long-term testing. Possible reasons also include the slight dissolution of Ir catalyst and the SiO_2 support.

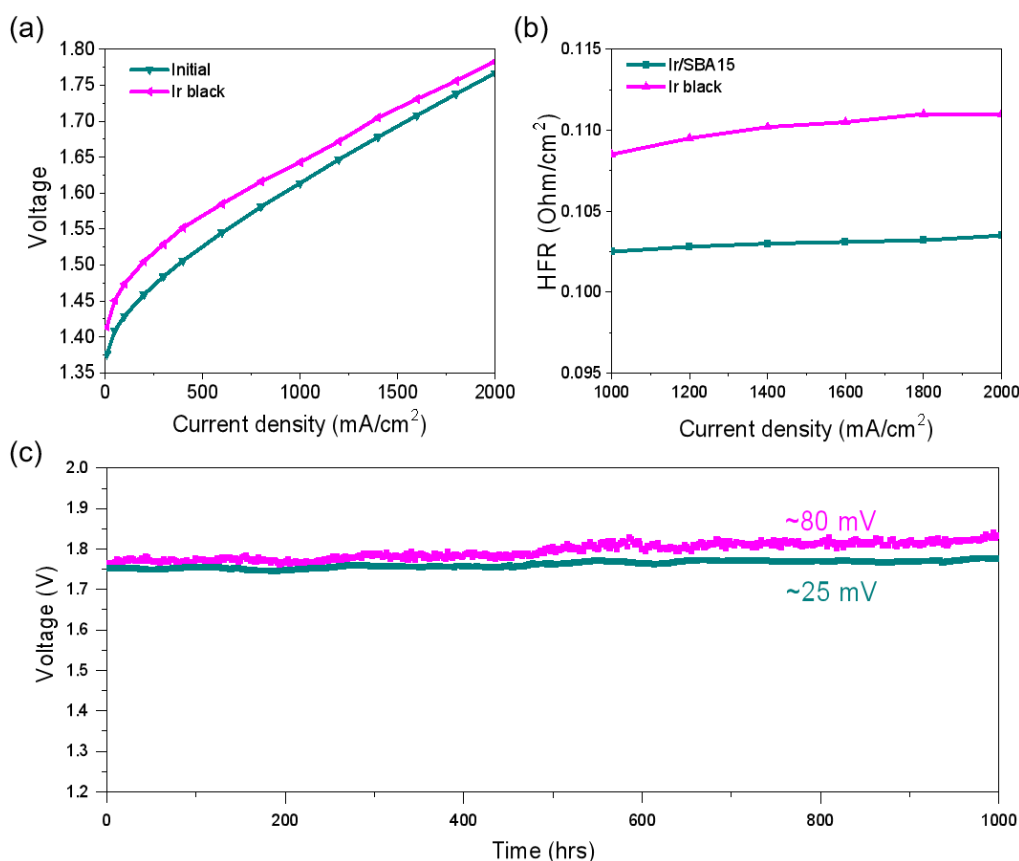


Figure 4. The electrolyzer performance (a), HFR (b), and 1000 hrs durability (c) for the electrolyzer with Ir/SBA15 and Ir black catalyst.

4. Conclusion

Iridium nanoparticles were uniformly deposited onto commercial, high-surface-area SiO_2 to develop a highly active and stable catalyst for oxygen evolution reaction (OER) applications. The formation of conductive iridium na-

nochains, along with the reduction in particle size, significantly enhanced OER activity by increasing the electrochemically active surface area and facilitating more efficient electron transport. These improvements are directly translated to enhanced performance in proton exchange membrane (PEM) electrolyzers. Electrochemical characterization revealed that the Ir/ SiO_2 composite not only exhibited excellent OER activity but also maintained strong cycling durabil-

ity, indicating good long-term operational stability under harsh conditions. These findings suggest that the tailored Ir/SiO₂ structure is a promising catalyst design for durable and efficient PEM water electrolysis. However, scaling up Ir/SBA15 catalysts for PEM electrolyzers faces key challenges, including the high cost and limited scalability of SBA15 synthesis, poor electrical conductivity, and difficulties integrating with membrane electrode assemblies. These limitations must be addressed through support modification, improved fabrication methods, or alternative, more scalable support materials.

Abbreviations

OER	Oxygen Evolution Reaction
PEM	Proton Exchange Membrane
TEM	Transmission Electron Microscopy
SBA15	Mesoporous Silica
Ir	Iridium
HER	Hydrogen Evolution Reaction
MEAs	Membrane Electrode Assemblies
XRD	X-ray Diffraction
ATO	Antimony Doped Tin Oxide
CCMs	Catalyst-Coated Membranes
XPS	X-ray Photoelectron Spectroscopy
HFR	High Frequency Resistance
RDE	Rotating Disk Electrode
CV	Cyclic Voltammetry

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Conflicts of Interest

The authors declare no conflicts of interest.

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