Iridium-Doped N-Rich Mesoporous Carbon Electrocatalyst with Synthetic Macrocycles as Carbon Source for Hydrogen Evolution Reaction

Xin Xiao, Hao Zhang, Ying Xiong,∗ Feng Liang,∗ and Ying-Wei Yang∗

Utilizing supramolecular synthetic macrocycles with distinct porous structures and abundant functional groups as a precursor for metal-doped carbon electrocatalysts can endow the resulting materials with great potential in electrocatalysis. Herein, iridium-doped electrocatalysts (CBC-Ir), using a synthetic macrocycle named cucurbit[6]uril as the carbon source precursor, are designed and prepared. Interestingly, owing to the numerous N-containing backbone and unique porous structure from cucurbit[6]uril self-assembly, the newly designed catalysts CBC-Ir possess abundant N-doped and mesoporous structures without the need of additional N sources and templates. The catalysts exhibit superior catalytic performance toward the hydrogen evolution reaction with high Faradaic efficiency (91.5% and 92.7%), superior turnover frequency (2.1 and 0.69 H2 s−1) at the 50 mV overpotential, and only 17 and 33 mV overpotentials in acidic and alkaline conditions reaching the current density of 10 mA cm−2, better than the commercial Pt/C (28 and 43 mV). This work not only expands the application of supramolecular macrocycles in the water splitting field but also provides a new approach for preparing robust electrocatalysts.

1. Introduction

Hydrogen (H2), as the energy carrier possessing high gravimetric energy density and a zero-polluting combustion product, is considered as the cleanest energy resource and the best candidate for fossil fuel.[1,2] To date, the technology of electrochemical water splitting has captivated considerable interest as the best green and sustainable way to generate hydrogen.[3,4] Unfortunately, the application of this technology is still greatly hindered from the huge energy consumption resulting from electrode polarization effects and sluggish kinetics of hydrogen evolution reaction (HER).[5,6] Therefore, designing and developing electrocatalysts with high activity are still of the exceeding urgency and challenging. It is generally acknowledged that noble metals, such as Pt, Ir, and Ru, are regarded as the state-of-the-art electrocatalysts for HER with low overpotentials and Tafel slopes.[7–10] However, their high cost and poor stability greatly impede the industrial application. Recently, great works have proved that introducing the carbon source precursor as the support is an amenable way to meet the above obstacles. Since precursor can efficiently combine with metals to prevent the aggregation of metal particles during the calcination and the electronic structure and chemical bonding energy are tuned at the interface of metal/carbon source, this strategy can efficiently enhance the activity and stability of electrocatalysts.[12–18] Nitrogen-doped mesoporous carbon materials are ideal matrices due to their high surface area, fast mass transfer ability, abundant defect structures, low cost, and good catalytic activity.[19,20] However, the conventional synthetic method for nitrogen-doped mesoporous carbon is in need of additional nitrogen sources and enormous pre/post-treatments, such as hard/soft template methods, acid etching, and hydrothermal.[21–23] which caused extra costs and works.

Cucurbit[6]uril (CB[6]) as a class of pumpkin-shaped rigid supramolecular macrocycle is composed of six glycoluril (−C2H2N4O2−) monomers bridged by methylene (−CH2−) groups, which bears many functional entities, such as carboxyl and amide groups, and a hydrophobic cavity and has a high affinity toward an array of neutral and cationic species through hydrophobic and ion–dipole interactions.[24–28] CB[6] allows the formation of porous supramolecular self-assembly through the H-bonds interaction between the carbonyl oxygen and lateral C=−H groups, in which every 1D porous channel is consisted of six CB[6].[29,30] These unique features in conjunction with the synthetic easy, low cost, and good chemical and

Compared with the most widely used carbon source precursors involving graphene, carbon nanotube, metal–organic frameworks (MOFs), and other carbonaceous materials,[40–43] CB[6] shows certain advantages: i) the heteroatom N is introduced into carbon framework by self-doping without any additional N sources due to its extremely rich content of N; ii) the porous supramolecular self-assembly can produce ample in-plane mesoporous structures during the pyrolysis without any addition tedious pre/post-treatments;[30,44] iii) the synthetic procedure of CB[6] is simple, environmental friendly, low-cost, and high-yielding. These unique structural characteristics of CB[6] greatly boost the electrical transfer and enhance the electrocatalytic activity of catalysts,[45] and thus using supramolecular macrocycle CB[6] as a deal carbon source precursor to produce efficient electrocatalysts is promising. However, up till now, CB[6] as the carbon source precursor has only attracted slight interest in supercapacitors[46] and oxygen reduction reaction,[44,47] while has not yet received enough attention in the exploration of electrocatalysis in HER.

Here, we propose and develop iridium (Ir) nanocluster-doped N-rich mesoporous carbon electrocatalysts (CBC-Ir) based on supramolecular macrocycle CB[6] as the carbon source precursor and then first apply them in HER over a wide range of pH values. In consideration of the high affinity of CB[6] toward certain metal cations, Ir$^{3+}$ cation can be easily and uniformly combined with porous supramolecular self-assembly (CB[6]-Ir). Then, upon pyrolyzing under N$_2$ atmosphere, the ring of CB[6] is opened, and the metal cations are reduced to nanoclusters.[46–49] Finally, the iridium nanoclusters encapsulated N-rich mesoporous carbon catalysts, i.e., CBC-Ir, are successfully prepared. The synthetic route is clearly illustrated in Figure 1.

### 2. Results and Discussion

During the pyrolysis, the material structure and electrocatalytic performance can be affected by the employed temperature, therefore, a series of materials (CBC-Ir-T, T = 600, 700, 800, and 900 °C, respectively; the numbers are in correspondence to the employed temperature) were obtained by pyrolyzing the supramolecular complex precursors (CB[6]-Ir) at 600–900 °C under N$_2$. The prepared CBC-Ir-T was characterized by powder X-ray diffraction (PXRD), as shown in Figure 2a. The characteristic peaks at 40.7° and 47.3° belong to the (111) and (200) planes of Ir nanoclusters, respectively, and the Ir nanoclusters have a metallic Fm-3m structure (PDF card, No. 46-1044). Furthermore, the crystallinity degree of Ir nanoclusters in CBC-Ir-T gradually enhanced with the increasing calcination temperature from 600 to 900 °C, and both catalysts CBC-Ir-800 and CBC-Ir-900 have good crystallinity (Figure 2). Moreover, upon increasing the calcination temperature, the intensity ratio $I_D/I_G$ increased from 1.081 to 1.201, implying the abundant defects existing in the samples, which could efficiently improve the catalytic performance.[51] Relatively, for the catalysts CBC-Ir-800 and CBC-Ir-900, the enhancement in $I_D/I_G$ values is slight, suggesting the electrocatalysts own a relatively stable structure at 800 °C with the big carbon defects.[47] Meanwhile, no peak was observed in the range of 400–800 cm$^{-1}$, demonstrating the characteristics of dispersive and ultrafine for Ir nanoclusters in figure 1.
The pore structures of CBC-Ir-T were determined by the N\textsubscript{2} adsorption isotherms. All the CBC-Ir-T materials presented typical type-IV isotherms with a hysteresis loop, suggesting the mesoporous features of the materials (Figure 2c). These generated mesoporous structures endowed CBC-Ir-T high Brunauer–Emmett–Teller (BET) area and enough electrode/electrolyte interface to greatly promote the ion or charge accumulation during the HER\cite{47,52}. The BET area gradually increased with the pyrolysis temperature. However, the pore structure of matrix might be collapsed and the aggregation of Ir nanoclusters was also resulting from the high temperature calcination (900 °C). These decreased the BET area and porous diameter of CBC-Ir-900 (Figure 2d). Therefore, the CBC-Ir-800 showed higher BET than that of other catalysts. From the results of XRD, Raman, N\textsubscript{2} adsorption, and subsequent catalytic performances, 800 °C (CBC-Ir-800) was considered as the best calcination temperature for subsequent characterization.

Besides that, the effect of the added amount of Ir\textsuperscript{3+} on the HER activity of CBC-Ir-800-X (X = 0.3, 0.9, 1.2, and 2.4 mg, respectively; the number stands for the corresponding amount of Ir\textsuperscript{3+} added) was also investigated. The morphologies of CB[6] and CBC (CB[6] calcined at 800 °C) were examined by scanning electron microscopy (SEM). The rod structures of original CB[6] material could be clearly observed in the SEM images, and after pyrolysis at 800 °C, the resultant CBC materials still preserved the rod structures of CB[6], indicating the robustness of CB[6] macrocycles (Figure S1, Supporting Information). The transmission electron microscopy (TEM) images of CBC-Ir-800-X confirmed that Ir nanoclusters were uniformly distributed in the porous graphite-like matrix CBC and the Ir nanocluster contents of the CBC-Ir-800-X gradually increased with the addition of Ir\textsuperscript{3+} (Figure 3a; Figure S2, Supporting Information). Whereas, when the amount of Ir\textsuperscript{3+} was 2.4 mg, the aggregation of Ir nanoclusters in CBC-Ir-800-2.4 was clearly observed, which led to a low catalytic performance for the HER. The contents of Ir in CBC-Ir-800-X (X = 0.3, 0.9, 1.2, and 2.4) are ≈1.2, 2.5, 3.7, and 5.8 wt%, respectively, as determined by the inductively coupled plasma-optical emission spectrometry. Subsequent HER performance evaluation disclosed that the catalyst CBC-Ir-800-1.2 has the highest catalytic activity among the four catalysts. Thus, CBC-Ir-800-1.2 was used as the optimized catalyst to implement the following research.

The crystalline structure of Ir nanoclusters in CBC-Ir-800-1.2 was further studied by high-resolution TEM (HR-TEM; Figure 3b). The ordered and clear lattice fringes of CBC-Ir-800-1.2 revealed the lattice distances of 0.221 and 0.192 nm in correspondence to the (111) and (200) crystal planes of Ir nanoclusters, respectively. Additionally, to reveal the composition and valence states of CBC-Ir-800-1.2, X-ray photoelectron spectroscopy (XPS) was measured. The C, N, O, and Ir were presented in CBC-Ir-800-1.2 as confirmed by the survey pattern, suggesting that N was successfully introduced into the carbon framework by self-doping approach (Figure S4a, Supporting Information). Specifically, the N 1s spectra of the CBC-Ir-800-1.2 could be deconvoluted into four types of nitrogen species including pyridinic N (397.7 eV), pyrrolic N (399.2 eV), graphitic N (400.5 eV), and oxidized N (402.1 eV), respectively (Figure 3c)\cite{44}. Besides, the relative contents of four types of N were also analyzed. Compared with the pure CBC, the relative content of pyridinic N for CBC-Ir-800-1.2 was increased from 22.89% to 25.65% and from 20.39% to 22.52% for pyrrolic N, meanwhile, the active sites of pyridinic N and pyrrolic N can interact with Ir nanoclusters by electron coordination to generate Ir–N and result in a change of N 1s binding energy (Figure 3c)\cite{53}. The presence of pyridinic

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**Figure 2.** a) PXRD patterns of CBC, CBC-Ir-600, CBC-Ir-700, CBC-Ir-800, CBC-Ir-900, and PDF#46-1044 (Ir). b) Raman spectra, c) nitrogen adsorption isotherm, and d) pore diameter distribution curves of CBC-Ir-600, CBC-Ir-700, CBC-Ir-800, and CBC-Ir-900.
N and pyrrolic N in CBC-Ir-800-1.2 can facilitate the fast charge transfer kinetics by interacting with H⁺, making the adsorption of H₂ easier. And graphitic N plays a critical role in electronic transmission.[44,50,54] Furthermore, the chemical state of Ir existing in CBC-Ir-800-1.2 was also measured by the high-resolution spectra of Ir 4f (Figure 3d). The Ir 4f peaks at 60.6 and 63.8 eV were attributed to the 4f \( \text{7/2} \) and 4f \( \text{5/2} \) of Ir\(^{0}\), whereas the peaks located at 62.1 and 65.8 eV were assigned to 4f \( \text{7/2} \) and 4f \( \text{5/2} \) of Ir\(^{4+}\), confirming the coexistence of metallic Ir and Ir\(^{4+}\).[50,55]

Inevitably, the concentration of proton or hydroxide can affect the catalytic performance during the electrocatalytic process, and an excellent electrocatalyst is expected to work effectively within a wide pH range. Therefore, in this work, the intrinsic HER performances of CBC-Ir catalysts were explored in N₂-saturated acidic (0.5 m H\(_2\)SO\(_4\)) and basic (1.0 m KOH) conditions. The polarization curves of CBC-Ir-T in Figure 4 suggested that the CBC-Ir-800 outperformed other catalysts with different calcination temperatures. Specifically, the CBC-Ir-800 material only requires the over-potentials (\( \eta \)) of 17 and 33 mV to deliver a 10 mA cm\(^{-2}\) current density in both 0.5 m H\(_2\)SO\(_4\) and 1.0 m KOH solutions, which were much lower than CBC-Ir-600 (151, 158 mV), CBC-Ir-700 (35, 86 mV), and CBC-Ir-900 (23, 49 mV). According to aforementioned XRD and N\(_2\) adsorption analysis, we deduced that the excellent HER activity of CBC-Ir-800 can be explained by its good crystallinity of Ir nanoclusters as well as the high BET area. However, the Ir nanoclusters can further aggregate and grow at a calcination temperature of 900 °C, resulting in the decrease of catalytic performance. Hence, 800 °C was used as the optimum calcination temperature.

Additionally, the electrochemically active surface area (ECSA) of catalysts was calculated, which was evidenced by a

![Figure 3. TEM images of a) CBC-Ir-800-1.2 (inset shows the corresponding particle size distribution of Ir). b) HR-TEM of CBC-Ir-800-1.2. c) High-resolution XPS N 1s of CBC and CBC-Ir-800-1.2. d) Ir 4f spectra of CBC-Ir-800-1.2.](image)

![Figure 4. HER polarization curves of CBC, CBC-Ir-600, CBC-Ir-700, CBC-Ir-800, and CBC-Ir-900 in the solutions of a) 0.5 m H\(_2\)SO\(_4\) and b) 1.0 m KOH at a scan rate of 3 mV s\(^{-1}\).](image)
double-layer capacitance (C_{dl}) on the basis of cyclic voltammetry (CV). Among the four catalysts CBC-Ir-800-X (X = 0.3, 0.9, 1.2, and 2.4), the catalyst CBC-Ir-800-1.2 was superior to the other three materials no matter in acidic (0.5 M H_2SO_4) or alkaline (1.0 M KOH) conditions with lower overpotentials as well as higher C_{dl} (Figure S5, Supporting Information). Therefore, the catalyst CBC-Ir-800-1.2 was further proved to be the best catalytic material.

In order to further confirm the advantages of CB[6] as the carbon source precursor in the catalysts, the Ir nanoclusters doped with other carbon source precursors including carbon nanotubes (CNT-Ir-800-1.2) and graphene oxide (GO-Ir-800-1.2) were synthesized using the same method as CBC-Ir-800-1.2. To eliminate the influence of other metals of MOF for catalytic performance, we selected ZIF-8, a representative of MOF material, as the precursor material, which was calcinated at 900 °C under N_2 to remove the zinc ions. The Ir nanocluster-doped mesoporous catalyst (ZIF-Ir-900-1.2) was obtained. Compared with CNT-Ir-800-1.2 (1.94 nm), GO-Ir-800-1.2 (6.26 nm), and ZIF-Ir-900-1.2 (5.25 nm), the CBC-Ir-800-1.2 material has the smaller particle size with an average size of Ir nanoclusters of 1.74 nm, as shown in Figure 3a and Figure S3 in the Supporting Information. The CB[6] possessing many carbonyl and amide groups is attractive for Ir^{3+} binding through an ion–dipole effect, which is helpful for dispersing Ir^{3+} in CB[6] self-assembly to form uniformly dispersed Ir sites and efficiently avoiding the particle gathering in the pyrolyzed samples.\textsuperscript{30,46} Besides, the BET surface areas were 558.11, 116.68, 211.71, and 885.97 m\textsuperscript{2} g\textsuperscript{-1} for CBC-Ir-800-1.2, CNT-Ir-800-1.2, GO-Ir-800-1.2, and ZIF-Ir-900-1.2, respectively. The corresponding pore diameter and total pore volume of CBC-Ir-800-1.2 were measured to be 4.61 nm and 0.994 cm\textsuperscript{3} g\textsuperscript{-1} (Table S1, Supporting Information) by the Barrett–Joyner–Halenda (BJH) method. Taking into account the high specific surface area, big pore diameter and small Ir nanoclusters, the CBC-Ir-800-1.2 catalyst was expected to exhibit good HER performance.

The electrocatalytic behaviors of the catalysts CBC-Ir-800-1.2, CNT-Ir-800-1.2, GO-Ir-800-1.2, and ZIF-Ir-900-1.2 were also evaluated. As expected, in comparison with CNT-Ir-800-1.2 (48.3/169.1 mV), GO-Ir-800-1.2 (90.3/221.7 mV), ZIF-Ir-900-1.2 (39.3/149.1 mV), and commercial Pt/C (28/43 mV), the CBC-Ir-800-1.2 material exhibited an optimal HER activity in Figures 5a and 6a, displaying a low overpotential of 17 and
33 mV to give a current density of 10 mA cm\(^{-2}\) in both 0.5 m H\(_2\)SO\(_4\) and 1.0 m KOH electrolytes on three-electrode cell system. The corresponding Tafel slopes of different materials were also extracted to explore the underlying reaction kinetics (Figures 5b and 6b). The Tafel slopes of CBC-Ir-800-1.2 (17.9 and 42.1 mV dec\(^{-1}\)) were smaller than that of CNT-Ir-800-1.2, GO-Ir-800-1.2, and ZIF-Ir-900-1.2, revealing a more favorable electrocatalytic kinetics for HER. Additionally, the CBC-Ir-800-1.2 displayed a comparable Tafel slope with Pt/C (16.7 and 38.5 mV dec\(^{-1}\)), suggesting that the process of HER follows the Volmer–Tafel mechanism, and the rate-determining step (RDS) is the adsorbed hydrogen atoms (Tafel)\(^{[56]}\).

In order to better understand the intrinsic HER activity of catalysts, the \(C_{dl}\) was measured in both solutions of 0.5 m H\(_2\)SO\(_4\) and 1.0 m KOH. The CBC-Ir-800-1.2 possesses the largest \(C_{dl}\) (91.26 mF cm\(^{-1}\)), which is 31, 16, 2.7, and 1.6 times larger than that of GO-Ir-800-1.2, CNT-Ir-800-1.2, Pt/C, and ZIF-Ir-900-1.2 in 0.5 m H\(_2\)SO\(_4\) solution (Figure 5c). Furthermore, the same results were also achieved in 1.0 m KOH with a highest \(C_{dl}\) of 76.14 mF cm\(^{-1}\) for CBC-Ir-800-1.2 (Figure 6c). The large \(C_{dl}\) of CBC-Ir-800-1.2 can be mainly attributed to the abundant N and ultrasmall Ir nanoclusters doping as well as the unique mesoporous structures of macrocycle CB[6], which generated more catalytically interfaces and active sites thus could efficiently boost charge/mass transfer, resulting in the excellent HER activity. Through the Nyquist plots, the electrochemical impedance spectroscopy (EIS) was measured to investigate the interfacial electron transfer dynamics of catalysts (Figures 5d and 6d). The semicircle diameters of the Nyquist plots represent the charge-transfer resistance (\(R_{ct}\)) of catalysts, which are related to the electrocatalytic kinetic. The CBC-Ir-800-1.2 possessed a much smaller resistance of 5.35 and 10.97 \(\Omega\) than that of CNT-Ir-800-1.2 (\(R_{ct} = 6.28\) and 11.38 \(\Omega\)), GO-Ir-800-1.2 (\(R_{ct} = 6.53\) and 14.69 \(\Omega\)), and ZIF-Ir-900-1.2 (8.51 and 15.11 \(\Omega\)) in both acid and the alkaline conditions, indicating a favorable charge transport in the interface of the CBC-Ir-800-1.2 and the electrolyte.

The mass specific activities were performed to further verify the high intrinsic HER activity of CBC-Ir-800-1.2. Figures 5e and 6e reveal that CBC-Ir-800-1.2 possessed high mass...
specific activities with 306 and 218 mA mgIr\(^{-1}\) at the overpotential of 15 mV in both of acidic and alkaline solutions, which are 15.3 and 8.1 times higher than Pt/C. In addition, the turnover frequency (TOF) values of CBC-Ir-800-1.2, which reflects the intrinsic catalytic activity, are calculated to be 2.1 and 0.69 H\(_2\) s\(^{-1}\), respectively, surpassing the Pt/C with 0.32 and 0.12 H\(_2\) s\(^{-1}\) in acid and alkaline conditions at an overpotential of 50 mV. Moreover, the Faradaic efficiency of CBC-Ir-800-1.2 showed up to 91.5% and 92.7% in 0.5 M H\(_2\)SO\(_4\) and 1.0 M KOH (Figures 5f and 6f). Once again, these results suggest a higher intrinsic HER activity of CBC-Ir-800-1.2.\(^{[57]}\) Notably, the CBC-Ir-800-1.2 material using supramolecular macrocycle CB[6] as carbon source precursor exhibited superior HER activities than that of the most recent reported electrocatalysts with different carbon source precursors including g-C\(_3\)N\(_4\), melamine, carbon black, and MOF (Table S2, Supporting Information). The superiority of CB[6] as the carbon source was further proved.

The stability of catalysts as another important property of electrocatalyst was also determined by using the CV scanning 1000 cycles and chronopotentiometry (V–t) method in the electrolytes of 0.5 M H\(_2\)SO\(_4\) and 1.0 M KOH. Compared with GO-Ir-800-1.2, CNT-Ir-800-1.2, and ZIF-Ir-900-1.2, the polarization curves of CBC-Ir-800-1.2 showed slighter change after 1000 CV cycles. Moreover, the V–t curve further confirmed the high stability of CBC-Ir-800-1.2 (Figures 7 and 8).

Interestingly, besides the Ir-related systems, the macrocycle CB[6] can also be employed as a general precursor material to prepare other noble metal-doped N-rich mesoporous carbon electrocatalysts for highly efficient HER. In this work, we also synthesized the Ru- and Pt-loaded catalysts CBC-Ru-800 and CBC-Pt-800, and then performed their HER performance in 0.5 M H\(_2\)SO\(_4\) and 1.0 M KOH (Figures S10–S13, Supporting Information). As expected, both of the electrocatalysts exhibited excellent HER properties with low overpotentials (51 and 37 mV for CBC-Ru-800, and 24 and 98 mV for CBC-Pt-800), high active surface areas (55.16 and 48.77 mF cm\(^{-2}\) for CBC-Ru-800, and 87.54 and 68.24 mF cm\(^{-2}\) for CBC-Pt-800), fast charge transport, as well as high stability in both acid and alkaline electrolytes.

Figure 7. HER polarization curves of a) CBC-Ir-800-1.2, b) Pt/C, c) CNT-Ir-800-1.2, d) GO-Ir-800-1.2, and e) ZIF-Ir-900-1.2 before and after 1000 CV, as well as f) the chronopotentiometry (V–t) curve of CBC-Ir-800-1.2 during HER at a constant current density of 10 mA cm\(^{-2}\) in 0.5 M H\(_2\)SO\(_4\).
3. Conclusion

In summary, the supramolecular macrocycle \[\text{CB}[6]\] was selected as the carbon source precursor to fabricate a series of noble metal-doped N-rich mesoporous electrocatalysts. Taking advantage of the distinctive self-assembly porous structures and the high N contents of \[\text{CB}[6]\], the resulting catalysts exposed and created abundant catalytic active sites for HER catalytic activity. Upon optimizing the calcination temperature (800 °C) and the addition amount of Ir (1.2 mg), the resulting CBC-Ir-800-1.2 material displayed superior catalytic activity for HER with the over-potentials of 17 and 33 mV at the current density of 10 mA cm\(^{-2}\) and a high Faradaic efficiency of 91.5% and 92.7%, as well as high TOF values (2.1 and 0.69 H\(_2\) s\(^{-1}\)) both in acid (0.5 m \(\text{H}_2\text{SO}_4\)) and alkaline (1.0 m KOH) conditions, which exceeded the commercial Pt/C as well as most of the previously reported HER catalysts based on other carbon source precursors. Due to the facile synthesis procedure, abundant heteroatom doping skeletons, and attractive porous material structures, we strongly believe that various synthetic macrocycles can be further explored as the precursors to afford a wide variety of functional carbon materials for applications in many electrochemical fields, such as electrocatalysis, batteries, and supercapacitors.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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