In-Situ Growth of Few-Layered MoS2 Nanosheets on Highly Porous Carbon Aerogel as Advanced Electrocatalysts for Hydrogen Evolution Reaction

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ABSTRACT: Molybdenum disulfide-based hybrids, acting as cost-effective and acid-stable electrocatalysts for hydrogen evolution reaction (HER), have been developed fast for providing sustainable hydrogen energy in recent years. Herein, few-layered molybdenum disulfide (MoS2) nanosheets/carbon aerogel (CA) hybrids were successfully obtained through the combination of sol−gel process, aging, freeze-drying, high temperature carbonization, and solvothermal reaction. CA with highly continuous porosity and high specific surface area is used as a matrix material for construction of hierarchical MoS2/CA hybrids where few-layered MoS2 nanosheets are uniformly covered on a CA surface. In this heterostructured system, CAs not only provide three-dimensional (3D) conductive pathway for fast transportation of electrons and ions, but also offer highly active regions for the growth of MoS2, greatly preventing the aggregation of MoS2 nanosheets. Due to the rationally designed hybrids with 3D porous nanostructures, the as-prepared MoS2/CA hybrids with optimized MoS2 content exhibit enhanced catalytic performance for electrocatalytic HER with a low onset potential of −0.14 V, large current density, and excellent stability.

KEYWORDS: Molybdenum disulfide, Carbon aerogels, Hydrogen evolution reaction

INTRODUCTION

More and more scientific works have been committed to search for clean and renewable energy alternatives due to the severe stress from global environmental pollution and the energy crisis derived from excessive consumption of fossil fuels. Among various energy storage methods, the water splitting reaction excited either by light or electricity for renewable hydrogen energy has attracted tremendous attention because of its cleanliness and potentially low cost. The essential step in water electrolysis is the hydrogen evolution reaction (HER), in which hydrogen is generated by electrocatalytic reduction of hydrogen ions. Platinum (Pt) and its alloys are very active catalysts for HER owing to their highly efficient energy conversion ability and low overpotential. However, the low natural reserves and expensive cost of Pt- and Pt-based alloys hamper their commercial application in electrochemical hydrogen generation. Transition metal dichalcogenides (TMD) materials, for instance, MoS2, WS2, MoSe2, WSe2, and VSe2, are developed as potential alternatives of Pt-group electrocatalysts for HER due to their constrained electrons within two-dimensional (2D) layers.

As a typical 2D TMD layered material, MoS2 shows graphene-like structure, in which molybdenum atoms are sandwiched between two layers of sulfur atoms. Recently, researchers have found that MoS2 can be a promising electrocatalyst for HER. Liu et al. prepared 2D MoS2 nanosheets from commercial MoS2 powder via liquid exfoliation and ultrasonication. The obtained MoS2 nanosheets exhibited extraordinary HER electrocatalytic performance, with onset potential lowered to −0.12 V. Previous works show that the defective sulfur (S) edges in MoS2 nanosheets have excellent electrocatalytic activity, which favors the HER process by decreasing the overpotentials and increasing the current densities. However, the basal planes of MoS2 are catalytically inert. Therefore, MoS2 nanosheets with small size and few stacked layers have better electrocatalytic activity because of the existence of more exposed sulfur edge sites. Until now, there are many kinds of preparation methods for the synthesis of nanosized MoS2, such as chemical vapor deposition, electro-
chemical deposition, hydrothermal reaction, and inverse micelle method. However, another obstacle for the practical application of MoS$_2$ nanosheets in the HER field is their poor conductivity. Therefore, increasing the conductivity of MoS$_2$ nanosheets while maintaining their nanosize is the key challenge to realize the practical application of MoS$_2$ nanosheets in HER. In this regard, preparation of uniformly distributed and edge-rich MoS$_2$ nanosheets on a conductive substrate is an effective strategy to enhance their catalytic activity for HER.

Carbon materials, including graphene, carbon nanofibers (CNFs), carbon nanotubes (CNTs), activated carbon, carbon papers, and so on, are ideal substrates for loading MoS$_2$ to advance their electrocatalytic performance due to the excellent conductivity and stability of these carbon materials.$^{23-40}$ MoS$_2$/carbon hybrids have enormous advantages, such as great varieties, high surface-to-volume ratio, tunable molecular structures, and good stability in a harsh environment. For example, Dai et al.$^{31}$ first reported few-layer MoS$_2$ nanosheets with abundant exposed sulfur edges stacked on reduced graphene oxide (rGO) sheets by a selective solvothermal method. In the MoS$_2$/rGO hybrids, rGO offers the conductive path for electron transfer between the catalyst and electrode, and provides active sites for the growth of MoS$_2$, which prevents the aggregation of MoS$_2$ and enhances the exposure of S-edges. Thus, the obtained MoS$_2$/rGO hybrids show excellent electrocatalytic performance, and the onset overpotential is low to $-0.1$ V along with a decreased Tafel slope (41 mV/decade). Du et al.$^{42}$ reported a novel synthesis of 2D MoS$_2$ with single layer nanosized nanoplatelets and S-edge-rich by hybridization with one-dimensional (1D) CNFs. The designed hybrids exhibit a decreased overpotential of 300 mV at high current density of 80.3 mA/cm$^2$ and a low Tafel slope of 42 mV/decade. Wang et al.$^{43}$ prepared low crystalline MoS$_2$ nanosheets coated CNTs which exhibited enhanced catalytic activity for HER. Among various carbon materials, carbon aerogel (CA) with 3D interconnected network and unique properties, including highly porous structure, large surface area, and great electron transport performance, is an ideal substrate for MoS$_2$ loading and can be used in energy area.$^{44-46}$ To our best knowledge, the nanocomposites of MoS$_2$ nanosheets and CAs have not been previously applied in the field of HER.

In this work, a novel and facile strategy is developed for the fabrication of 3D CA supported MoS$_2$ nanosheets with the combination of sol–gel process, high temperature carbonization, and solvothermal reaction. CA acting as the 3D conductive substrate can not only prevent the aggregation of MoS$_2$ and enhance the exposure of active S-edges, but also improve the conductivity of the MoS$_2$/CA hybrids, thus facilitating electron transfer during the electrocatalysis process. Morphological characterizations show that few-layered MoS$_2$ nanosheets with abundant edges are vertically grown on the surface of CA substrate uniformly. Owing to the highly exposed edge sites and relatively low aggregation, the obtained MoS$_2$/CA hybrids exhibit excellent electrocatalytic properties, with a low onset potential of $-0.14$ V, large current density, and excellent stability, making it a potential electrocatalyst for HER.

## EXPERIMENTAL SECTION

**Materials.** Pyromellitic dianhydride (PMDA), N,N-dimethylacetamide (DMAc), triethylamine (TEA, 99%), 4,4’-oxidianiline (ODA), 30% H$_2$O$_2$, 98% H$_2$SO$_4$, KMnO$_4$, 37% HCl, N,N-dimethylformamide (DMF), ammonium molybdate, and thiourea were obtained from Sinopharm Chemical Reagent Co., Ltd. All the above reagents were used as received without any treatments. Natural graphite powder (325 mesh) was supplied by Alfa-Aesar (Ward Hill, MA) and used without further treatments. All other chemicals were obtained from Aladdin Chemical Reagent, Co., Ltd., and used as received.

**Preparation of MoS$_2$/CA Hybrids.** Polyimide (PI)-based CAs (derived from graphene cross-linked PI aerogels) were synthesized according to our methods reported previously.$^{47}$ The preparation of MoS$_2$/CA hybrids is shown in Scheme 1. First of all, the bulk CAs were smashed into powder by continuous ball-milling at 500 rpm for 4 h. MoS$_2$/CA hybrids with various MoS$_2$ amounts were synthesized via a one-step solvothermal method according to Xie’s method.$^{48}$ In brief, proper contents of ammonium molybdate and thiourea with a molar ratio of 1:2 were added to 60 mL water, and this was followed by addition of a certain amount of CAs. Then, the mixture was well-mixed by magnetic stirring for 2 h at room temperature. The obtained dispersion was transferred to a 100 mL Teflon stainless-steel autoclave and reacted at the temperature of 200 °C for 12 h. The precipitates were obtained through centrifugation at 12 000 rpm for 10 min, and then were washed with DI water and anhydrous ethanol several times and finally dried under vacuum at 80 °C for 6 h. Afterward, the samples were calcined at 300 °C for 2 h with a heat rate of 2 °C min$^{-1}$ under N$_2$ atmosphere. Finally, the MoS$_2$/CA hybrids with the initial CA/Mo weight ratio of 1:2, 1:4, and 1:8 were obtained and denoted as MoS$_2$/CA-2, MoS$_2$/CA-4, and MoS$_2$/CA-8, respectively. For comparison, pure CA and pure MoS$_2$ were prepared under the same conditions.

**Characterization.** The microstructures of the obtained samples were characterized by field emission scanning electron microscopy (FESEM) (Ultra 55, Zeiss) at 5 kV acceleration voltage. The chemical composition was characterized by the energy dispersive X-ray spectroscopy (EDX). Transmission electron microscopy (TEM) and high resolution transmission electron microscopy (HRTEM) observations were conducted with JEOL JEM 2100 TEM under 200 kV acceleration voltage. X-ray diffraction (XRD) patterns were performed on an X’Pert Pro X-ray diffractometer with Cu K$_\alpha$ radiation ($\lambda = 0.1542$ nm) under a current of 40 mA and a voltage of 40 kV with 2θ ranges from 5° to 80°. X-ray photoelectron spectroscopy (XPS) analyses were performed with a VG ESCALAB 2201-XL device, and all XPS spectra were corrected using C 1s line at 284.5 eV. In addition, the curve fitting and background subtraction were accomplished using XPS PEAK41 software. In order to calculate the mass content of MoS$_2$ nanosheets in the hybrids, thermogravimetric analysis (TGA) was used under air flow from 100 to 700 °C at a heating rate of 20 °C/min.

**Electrochemical Measurements.** Prior to all the experiments of hydrogen evolution performance, glassy carbon electrodes (GCE) of 3 mm in diameter were preprocessed according to the previous report.$^{21}$
Typically, the working electrode was prepared as follows. A 2 mg portion of MoS$_2$/CA hybrid was dispersed in 1 mL of a mixed solvent (DMF and deionized water by a volume ratio of 1:1) containing 20 μL 5 wt% nafion. Then, the mixture was sonicated at least 15 min in order to obtain the homogeneous suspension. Finally, 10 μL of the homogeneous mixture was dropped onto GCE to form MoS$_2$/CA hybrid modified GCE. The required loadings of the electrocatalyst were adjusted by repeatedly adding 5 μL of the obtained MoS$_2$/CA hybrid slurry.

All electrochemical catalytic research was carried out by a CHI 660D electrochemical workstation (Chenhua Instruments Co, Shanghai, China) at room temperature. The hydrogen evolution performance tests were performed in the electrolyte solution of 0.5 M H$_2$SO$_4$. For a standard typical three-electrode cell, the different electrocatalyst modified GCE was applied as the working electrode, with saturated calomel electrode (SCE) as the reference electrode and Pt wire as counter electrode, respectively. In our electrochemical tests, all the potentials were calibrated to RHE according to the equation $E_{\text{RHE}} = E_{\text{SCE}} + (0.241 + 0.059 \, \text{pH}) \, \text{V}$. The electrocatalytic performance of MoS$_2$/CA hybrid toward HER was performed by linear sweep voltammetry (LSV) in nitrogen purged electrolyte solution, and the scan rate was 2 mV/s. Electrochemical impedance spectroscopy (EIS) measurements were conducted in 0.5 M H$_2$SO$_4$ by applying an ac voltage in the frequency range between 100 kHz and 10 mHz with 5 mV amplitude.

**RESULTS AND DISCUSSION**

**Morphology and Structures of MoS$_2$/CA Hybrids.** The typical structure of CA and CA particles after ball-milling is shown in Figure 1. As shown in Figure 1A, the obtained CA possesses high porosity, and the pore sizes range from dozens...
of nanometers to hundreds of nanometers. These porous structures can offer a 3D conductive substrate, which is beneficial for ion and electron transport. Irregular structures with many sharp edges and sizes of hundreds of nanometers are observed for CA particles (Figure 1B). The irregular structure of CA particles favors the growth of MoS2 nanosheets so that they can not only prevent the agglomeration of MoS2 nanosheets, but also increase the exposure of the active MoS2 edges, thus highly improving the catalytic performance for HER. Specific surface area and porous structure of the obtained CA are characterized by nitrogen physisorption isotherms (Figure 1C,D). The specific surface area of CA is 978 m²/g, and the isotherm curve belongs to type IV with a hysteresis loop, indicating that the CA possesses a large quantity of mesopores. The pore size distribution (in the range 0−140 nm) measured by the Barrett−Joiner−Halenda method presents a relatively narrow distribution, which was centered at 15 nm. Therefore, the obtained CA with large surface area and high porosity is considered as a promising template for further construction of MoS2/CA hybrids with hierarchical nanostructures.

CA with different loading amounts of MoS2 nanosheets were prepared with the same procedure by adjusting the weight ratio of CA/Mo from 1:2, 1:4 to 1:8. After in-situ solvothermal reaction of CA powder in molybdenum salt solution, few-layered MoS2 nanosheets are evenly grown onto 3D conductive CA substrate (Figure 2A−C). By increasing the loading amount of molybdenum salt, more and more thin MoS2 nanosheets begin to form and densely grow on the CA particles. It is worth mentioning that MoS2 nanosheets are evenly and perpendicularly grown on the porous CA substrate when the weight ratio of molybdenum salt precursor to CA is 4:1. However, with increasing the weight ratio of molybdenum salt precursor to CA to 8:1, MoS2 nanosheets began to accumulate and stack together on CA substrate due to the limited growth space (Figure 2C). The EDX mapping analysis of the MoS2/CA-4 hybrid (Figure 2D) proves the coexistence and homogenous dispersion of C, Mo, S elements, further confirming that MoS2 nanosheets are evenly anchored on the surface of CA particles. In contrast, as shown in Figure 3, pure MoS2 prepared without adding CA particles consists of large microsized sheets, which are disorderly stacked together and aggregated into nanospheres. In addition, specific surface area and porous structure of the obtained pure MoS2 and MoS2/CA-4 hybrid is characterized by nitrogen physisorption isotherms, as shown in Figure S1. The specific surface area of MoS2/CA-4 hybrid is 107 m²/g, which is much larger than that of pure MoS2 (13 m²/g). The main reason is due to the special structure of CA with high surface area (978 m²/g) being able to offer more active sites for the growth of MoS2 nanosheets, which is beneficial for preventing the aggregation of MoS2 nanosheets. The pore size distribution of pure MoS2 and MoS2/CA-4 hybrid calculated by the Barrett−Joiner−Halenda method presents a relatively narrow distribution, which is centered at 4 nm. The high surface area and porous structure of MoS2/CA-4 hybrid is beneficial for electrolyte permeation and efficient ion diffusion, thus facilitating the HER electrocatalytic performance.

Morphology of MoS2/CA-4 hybrid is further confirmed by TEM observations (Figure 4). The irregular CA particles are clearly observed, and few-layered MoS2 nanosheets are evenly coated on CA substrate, which is in good accordance with SEM observations (Figure 2). From the HRTEM image in Figure 4B, 5−8 layers of MoS2 nanosheets can be clearly observed, and the interlayer spacing of MoS2 nanosheets is about 0.65 nm, which is in accordance with the (002) lattice of hexagonal MoS2.

The XRD patterns of pure CA, pure MoS2, and MoS2/CA-4 hybrid are shown in Figure 5. As for CA sample, the broad diffraction peak centered at 2θ = 26° and the weak diffraction peak at 2θ = 44° can be assigned to the (002) and (100) planes, respectively, revealing the low crystalline degree of CA. For pure MoS2 and MoS2/CA-4 hybrid, the diffraction peaks present similarly to each other, indicating that no additional crystallization behavior is introduced into the MoS2/CA-4 hybrid. In addition, all the diffraction peaks of pure MoS2 and MoS2/CA-4 hybrid can be indexed to the hexagonal MoS2 phase, which are in good accordance with the literature values (JCPDS: 00-037-1492). As shown in Figure 5, the MoS2/CA-4 hybrid shows sharp peaks at 2θ = 14.2°, 33.8°, and 59.3°, which can be indexed to (002), (100), and (110) planes of MoS2, respectively. To emphasize, the diffraction peak of (002) shifted from 2θ = 16.7° to 14.2° as compared to the standard hexagonal 2H-MoS2 structure, indicating an expanded inter-

![Figure 3](Image)

Figure 3. FESEM images of pure MoS2 at low (A) and high (B) magnifications.

![Figure 4](Image)

Figure 4. TEM (A) and HRTEM (B) images of MoS2/CA-4 hybrid.
layer. In addition, the (103) and (201) peaks at $2\theta = 39.8^\circ$ and $69.8^\circ$ can be weakly detected. Therefore, the XRD results suggest that MoS$_2$ has been successfully grown on the surface of CA.

Figure 6 shows the XPS spectra of the MoS$_2$/CA-4 hybrid. As shown in Figure 6A, the survey scan indicates that C, Mo, S, and O elements coexist in the MoS$_2$/CA-4 hybrid. The peak of C 1s spectrum is centered at 284.5 eV, which corresponds to sp$^2$ C (Figure 6B). High resolution Mo 3d spectrum (Figure 6C) shows characteristic peaks centered at 232.3 and 229.2 eV corresponding to Mo 3d$_{3/2}$ and Mo 3d$_{5/2}$ orbitals, suggesting that Mo in the MoS$_2$/CA-4 hybrid is in the Mo(IV) state. In addition, the binding energies of S 2p$_{1/2}$ and S 2p$_{3/2}$ orbitals were centered at 163.1 and 162.0 eV, indicating the existence of divalent sulfide ions (S$^{2-}$) (Figure 6D). The loading amounts of MoS$_2$ in the MoS$_2$/CA hybrids are calculated from the TGA curves (Figure 7), which is 21.6%, 38.4%, and 68.1% for MoS$_2$/CA-2, MoS$_2$/CA-4, and MoS$_2$/CA-8 hybrids, respectively.

**Electrochemical Performance of MoS$_2$/CA Hybrids.**

Generally speaking, an optimal HER catalyst is a material that could give the highest current at the lowest overpotential, as well as a low HER onset potential (i.e., the potential at which HER activity begins) comparable to that of Pt catalyst. The electrocatalytic performance of the MoS$_2$/CA hybrids for HER were carried out in the electrolyte solution of 0.5 M H$_2$SO$_4$ using a standard typical three-electrode cells. Typically, the hybrids with an optimized electrocatalyst loading weight of 20 $\mu$g were deposited on GCE. The polarization curves for all the hybrids were optimized, and commercial Pt/C catalysts, pure CA, and pure MoS$_2$ were also measured as reference (Figure 8). As shown in Figure 8A, either CA or pure MoS$_2$ exhibits no or
poor HER electrocatalytic performance due to the large onset overpotential and low current densities. All three MoS2/CA hybrids with different Mo/CA ratios do have good electrocatalytic activity, while MoS2/CA-4 hybrid exhibits the optimized electrocatalytic performance, with onset potential at approximately −0.14 V (vs RHE), and high current densities of 1.72 and 9.68 mA/cm² at overpotentials of 150 and 200 mV, respectively. The improved electrocatalytic HER activity for MoS2/CA hybrids suggests the synergistic effect between 3D conductive CAs and electroactive MoS2 nanosheets. As mentioned above, highly porous CA provides 3D conductive templates, which are conductive to reduce the diffusion path for ions and electrons. Besides, the distinctive structures of CA particles are able to offer many active sites for the homogeneous growth of MoS2 nanosheets and thus prevent the self-aggregation of MoS2 nanosheets. Furthermore, the irregular shape of CA particles can provide many sharp edges, maximizing the exposure of accessible active catalytic sites of MoS2 nanosheets. For MoS2/CA-2 hybrid, only sparse MoS2 nanosheets are interspersed on the surface of CA particles, leading to less electroactive sites for hydrogen evolution. In contrast, as for the MoS2/CA-8 hybrid, excess loading of MoS2 nanosheets could result in the aggregation of MoS2 nanosheets, limiting the exposure of MoS2 edges and electroactive sites. Therefore, with uniform distribution of MoS2 nanosheets and 3D conductive network of CA template, MoS2/CA-4 hybrids show synergistically improved catalytic performance for HER.

A Tafel slope is closely related to reaction path and adsorption type during the HER process. Therefore, it is always used to evaluate the catalytic effectiveness of catalysts. Usually, the Tafel curve derived from the polarization curves is obtained on the basis of fitting of the straight line part, and the Tafel slope is the slope of the fitting line. For the Tafel curve, the overpotential (η) and the relevant current density (j) are obtained from the LSV curves. The linear portions of the Tafel curve agree with the Tafel equation (η = b log(j) + a, where η is the overpotential, j is the current density, and b is the Tafel slope) at different overpotential ranges. The Tafel curves for pure MoS2, MoS2/CA-4 hybrid, and Pt, obtained from the LSV curves, are shown in Figure 8B. As calculated from Figure 8B, the Tafel slopes are ∼86, ∼59, and ∼31 mV/decade for pure MoS2, MoS2/CA-4 hybrid, and Pt, respectively. Compared to pure MoS2, the improved HER performance of the MoS2/CA-4 hybrid suggests a smaller activation energy for HER, which can be attributed to the effective hybridization of 3D electrical conductive CA and the homogeneous, nanosized, and S-edge-rich MoS2 nanosheets. According to the typical electrocatalytic mechanism in acidic aqueous for HER, the rate-determining step of the obtained pure MoS2 belongs to the Volmer reaction due to the intrinsic poor conductivity and low activity that arose from its microsize and disordered stacking. The evidently reduced slope for MoS2/CA-4 hybrid indicates that the hydrogen evolution takes place via the rapid Volmer reaction followed by a rate-determining Heyrovsky step.

Figure 9 shows the HER catalytic performance of different loadings of MoS2/CA-4 hybrid on GCE. As shown in Figure 9AB, the optimal loading of MoS2/CA-4 hybrid is 20 μg with current densities of 1.72 and 9.68 mA/cm² at overpotentials of 150 and 200 mV, respectively. For 30 and 40 μg loadings, the current densities at different overpotentials are lower than that
of 20 μg loading, which may be explained by the observation that excess loading of catalyst will increase the internal resistance and decrease the effective active sites.

In order to assess the electrode kinetics and electrical conductivity of the different electrocatalysts, EIS of pure CA, pure MoS2, and MoS2/CA hybrids were measured from 0.01 to 100 000 Hz. As can be seen from Figure 10, the Nyquist plots in Figure S3, all the catalytic current densities slightly fluctuated up and down due to the bubble formation in the process. Besides, with increasing the weight ratio of molybdenum salt precursor to CA, the current density reduced faster while MoS2/CA hybrid needs higher voltage to ensure the current density due to the decrease of conductivity with fewer CA particles.

**CONCLUSIONS**

In summary, a highly active electrocatalyst of MoS2/CA hybrid was fabricated where MoS2 nanosheets were grown uniformly on 3D mesoporous CA template by a simple solvothermal reaction. CA has abundant mesopores, a high surface area, and a highly conductive skeleton which provide a specific microenvironment and conductive pathways to accelerate the transportation of electrons and ions during the HER process. Besides, CA acting as conductive substrate can effectively prevent the aggregation of MoS2 nanosheets, and thus, the well-dispersed and edge-rich MoS2 nanosheets grown on CA template guarantee the full exposure of active edge sites. Therefore, the obtained MoS2/CA-4 hybrid possesses excellent catalyst performance for HER with a low onset potential of −0.14 V, large current densities (1.72 mA/cm2 at η = 150 mV; 9.68 mA/cm2 at η = 200 mV, relatively), and a small Tafel slope of 59 mV/decade. Besides, the MoS2/CA-4 hybrid shows long-term durability after 2000 cycles. Therefore, the highly electrocatalytically active CA supported nanosized MoS2 is a promising candidate for low cost electrocatalysts in hydrogen evolution field.

**ASSOCIATED CONTENT**

Supporting Information
The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acssuschemeng.5b00700.

N2 adsorption–desorption analysis, ac impedance spectroscopy of MoS2/CA-4 hybrid under different overpotentials, current–time responses of MoS2/CA hybrids, and LSV polarization curves for MoS2/CA-4 hybrid at different scan rates (PDF)

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