



Enhanced electrooxidation of urea using NiMoO₄·xH₂O nanosheet arrays on Ni foam as anode



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ABSTRACT

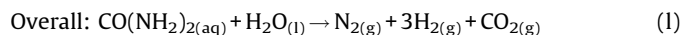
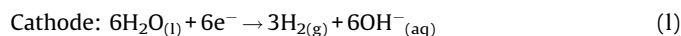
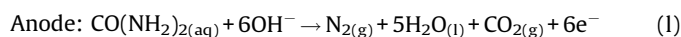
Urea electrolysis has shown great potential in hydrogen production and remediation of urea-containing wastewater and it is highly important to develop efficient urea oxidation electrocatalysts made from earth-abundant elements. Here we report the growth of NiMoO₄·xH₂O nanosheet arrays on Ni foam (NiMoO₄ NAs/NF) through a hydrothermal process. When directly used as a 3D catalytic anode for electrooxidation of urea in alkaline solutions, the NiMoO₄ NAs/NF exhibits high catalytic activity and stability. It achieves specific current density of 830 mA cm⁻² mg⁻¹ at 0.5 V at a scan rate of 10 mV s⁻¹, about 4.2 times enhancement compared to Ni(OH)₂ NAs/NF.

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1. Introduction

The depletion of fossil fuels and increased environmental concerns has triggered an urgent demand for searching clean and sustainable alternative energy sources [1]. Hydrogen is an abundant zero-emission fuel with great promise to replace fossil fuels in the future [2]. Electrolysis of water is one simple way to produce hydrogen. However, the thermodynamics cell voltage of 1.23 V for water electrolysis is much larger than 0.37 V required for urea electrolysis at standard conditions, and therefore, hydrogen production from urea electrolysis should be more energy-efficient [3]. Furthermore, compared with other gas/liquid hydrogen carriers, urea has a variety of unique properties such as stable, non-toxic, non-flammable and renewable [4–7]. On the other hand, urea-rich wastewater is largely produced from both animal excreta and industrial synthesis of urea [8–10]. Although urea is not directly toxic, its hydrolysis into ammonia leads to toxicity to both animal and marine life. Urea electrolysis has proven to be an effective technology to remedy urea-rich wastewater with the simultaneous production of highly pure hydrogen [8,11–13]. The

chemical reactions for urea electrolysis in alkaline media at electrodes are described by the following equations [14]:



However, an efficient catalyst for urea electrooxidation is usually required to achieve high current densities at low overpotentials. Ni is one interesting 3D metal with catalytic power for urea electrooxidation and recent years have witnessed the rapid development of Ni-containing electrocatalysts, including metallic Ni [3,6,8,15], nano-sized nickel [16], NiOOH [17], nickel hydroxide [18], Ni-Co bimetallic hydroxide [9], Ni(OH)₂ nanotubes on Ni framework [7], NiO nanosheets on 3D Ni foam [19], mesoporous NiCo₂O₄ [20], and graphene-Ni nanocomposites [21] etc.

Nitrogen and carbon dioxide gases are produced from the anode and hydrogen gas was generated from cathode during electrochemical process in alkaline media. Although Ni-based catalysts have shown superior catalytic activity over noble metals like Pt, Ir

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and Rh in alkaline media [8], degradation of the Ni catalysts leads to deactivated catalytic surface and thus decreased current density during urea electrooxidation [22]. Further incorporation of noble metals into nickel catalysts leads to enhanced urea electrooxidation current [22,23] but increases the cost of catalysts at the same time. As such, it is highly desired to develop efficient and stable urea electrooxidation catalysts made from earth-abundant elements.

Most of current catalysts are required to be effectively immobilized on electrode surfaces using a polymer binder like Nafion or polytetrafluoroethylene (PTFE) prior to electrochemical tests. Such polymer binder, however, generally increases the series resistance on one hand [24], and may also block active sites and inhibit diffusion on the other hand [25]. As a result, decreased catalytic activity is achieved. Our recent studies have shown that such issues can be solved by directly growing the catalysts on the current collectors for binder-free catalytic electrodes [26–28]. In this paper, for the first time, we demonstrate the development of $\text{NiMoO}_4 \cdot x\text{H}_2\text{O}$ nanosheet arrays on Ni foam (NiMoO_4 NAs/NF) as novel 3D catalytic anode for urea electrooxidation in alkaline solutions. This NiMoO_4 NAs/NF electrode exhibits high catalytic activity and stability with 4.2 times enhancement in specific current density compared to Ni(OH)_2 NAs/NF at 0.5 V.

2. Experimental

2.1. Materials

$\text{Ni(CH}_3\text{COO)}_2 \cdot 4\text{H}_2\text{O}$, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$, $\text{Ni(NO}_3)_2 \cdot 6\text{H}_2\text{O}$, urea and hexamethylenetetramine (HMT) were purchased from Beijing Chemical Reagent Co., Ltd. All the reagents were of analytical grade and used without further purification. The water used throughout all experiments was deionized (DI) water purified through a Millipore system. Prior to the synthesis, the Ni foam (NF) was cleaned by sonication in ethanol and DI water for 10 min, respectively.

2.2. Preparation of Ni(OH)_2 NAs/NF

A 35 ml aqueous solution containing 3.5 mmol $\text{Ni(NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 7 mmol HMT was stirred for 10 min, then transferred to a 50 mL Teflon-lined stainless-steel autoclave. Afterwards, the pre-treated NF ($2\text{ cm} \times 3\text{ cm}$) was immersed into the solution and hydrothermally treated at 100°C for 12 h and then cool down to ambient atmosphere. The substrates were taken out and cleaned by sonication in ethanol and DI water for several minutes to remove the loosely attached products on the surface, and then

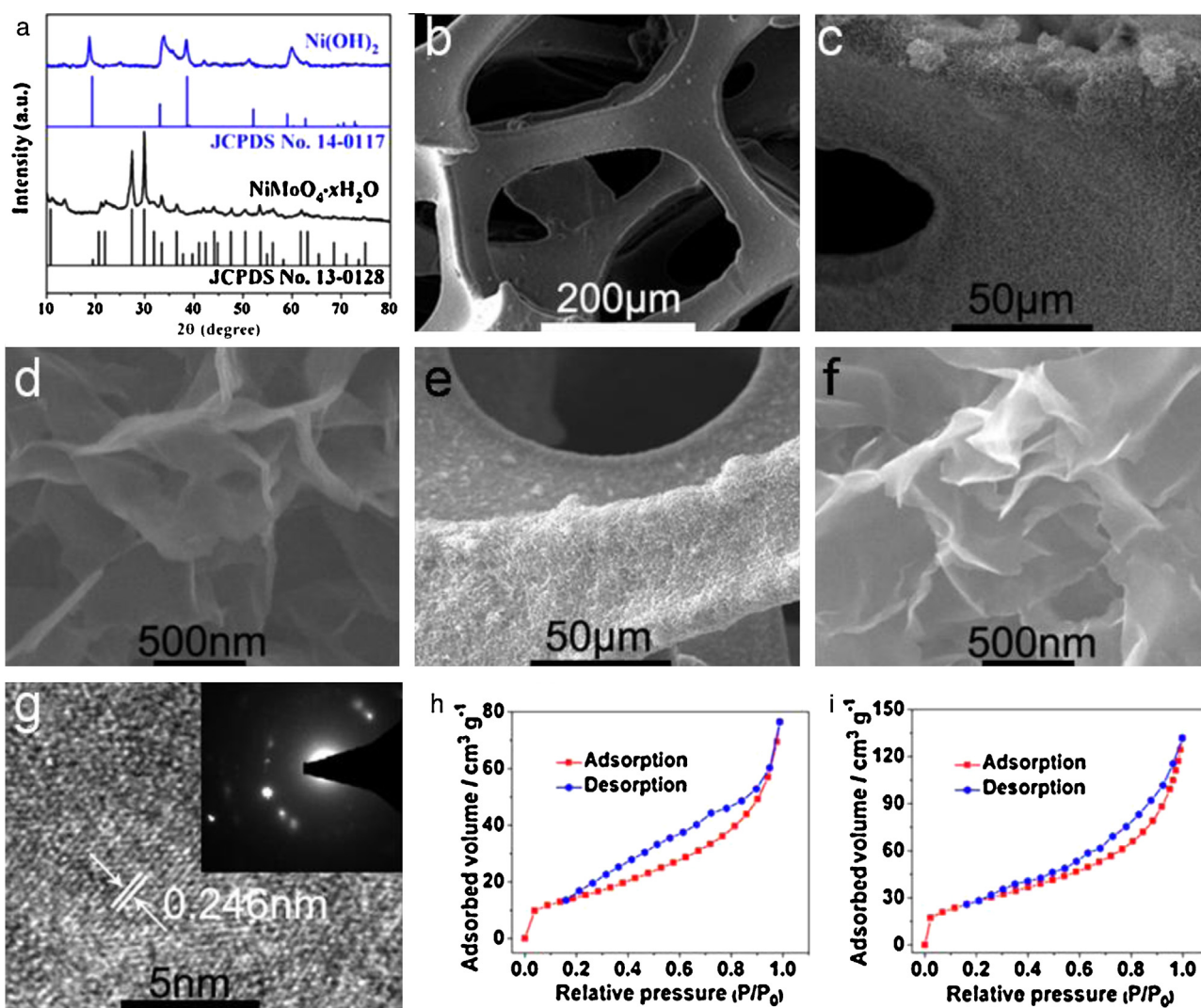


Fig. 1. (a) XRD patterns of Ni(OH)_2 and $\text{NiMoO}_4 \cdot x\text{H}_2\text{O}$ scratched down from Ni foam. SEM images of (b) bare Ni foam, (c, d) Ni(OH)_2 NAs/NF and (e, f) $\text{NiMoO}_4 \cdot x\text{H}_2\text{O}$ NAs/NF. (g) HRTEM image and SAED pattern (inset) taken from the $\text{NiMoO}_4 \cdot x\text{H}_2\text{O}$ nanosheet. Nitrogen adsorption/desorption isotherm plots of (h) Ni(OH)_2 and (i) $\text{NiMoO}_4 \cdot x\text{H}_2\text{O}$.

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