Morphology-controlled Synthesis of 1D Submicro-structured Polyoxomolybdate Towards Electrocatalytic Oxygen Reduction

DENG Zhong-hua, LV Gong-xuan*

(State Key Laboratory for Oxo Synthesis and Selective Oxidation, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000, China)

Abstract: 1D submicro-structured polyoxomolybdate submicro-wires, submicro-rods and nanobelts were synthesized by altering the pH value and reactants. Submicro-wires could be obtained by reacting $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ with sodium dodecyl benzene sulfonate (SDBS) at pH=6, and that for submicro-rods at pH=2. However, nanobelts could be synthesized by reacting $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ with NaCl at pH=6. In solution of pH=6, polyoxomolybdate should grow up submicro-wires in long micelles. At pH=2, not only more $Mo_3O_{10}^{2^2}$, but also short micelles formed by excessive H⁺ cations, which leaded to the submicro-rods. Without template SDBS, polyoxomolybdate may exist in stable nanobelts. Cyclic voltammetry results indicated that polymolybdate submicro-rods exhibited excellent activity for oxygen electroreduction in organic solvent. Because alcohol could react with H⁺ in the electrolyte, the activity of oxygen electroreduction over polymolybdate could be obviously promoted in the presence of alcohol. **Key words**: Polyoxomolybdate; 1D submicro-structured; Oxygen electroreduction; Alcohol promoted

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Direct methanol fuel cells (DMFCs) provide significant advantages, such as high energy density, low pollution, rapid start-up, and compactness over rechargeable batteries, and other types of fuelcells for portable applications^[1]. However, the DMFC performance is still hindered by several factors, including the high cost of the Pt-based electrocatalysts, the poor kinetics of both anode^[2], and cathode reactions^[3] and the crossover of methanol from the anode to the cathode through the proton exchange membranes^[4-5]. To solve the crossover problem, one strategy is the development of novel oxygen reduction reaction (ORR) electrocatalysts with high catalytic activity and methanol tolerance^[3-12]. Polyoxometalates (POMs), a large class of inorganic transition metal oxygen cluster compounds have attracted a great deal of interest as electrocatalysts for oxygen reduction reaction^[13-30].

Here, for the first time, we report a facile route to synthesize 1D NaNH₄Mo₃O₁₀ \cdot H₂O and obtained submicro-wires, submicro-rods, nanobelts, and the mixture of submicro-wires, nanobelts and microflowers by tuning the pH value, and reactants. Moreover, the 1D submicro-structured materials were used as electrocatalysts for ORR in organic solvent and alcohol can promote the activity of ORR.

1 Experimental

1.1 Synthesis of NaHH₄Mo₃O₁₀ · H₂O

1.41 g of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ was added into 10 mL solution containing 0.1 mol/L sodium dodecyl benzene sulfonate (SDBS) under magnetic stirring, then the obtained transparent solution was stirred for

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Biography: Deng Zhong-hua, born 1975, mile, Post Ph. D

^{*} Corresponding author: To whom correspondence should be addressed. E-mail:gxlu@lzb.ac.cn Tel./Fax: +86-931-4968178.

about 14 h at 25 °C. The white precipitate was filtered, and washed with deionized water for 6 times and then with absolute ethanol. Finally, the precipitate was dried at room temperature. The reaction conditions for the controlled synthesis of $NaNH_4Mo_3O_{10} \cdot H_2O$ submicrowires, submicro-rods, nanobelts, and the mixture is summarized in Table 1. The pH value of the reaction system was adjusted by HNO₃ solution (1.5 mol/L).

Sample	Reactants and ratios	pН	Template content	Temp.	Time	Morphology
		value	$/(\text{mol} \cdot L^{-1})$	$^{\circ}$ C	∕h	
S-1	Mo^a : SDBS = 8 : 1	6	0.10	25	14.0	submicro-wires
S-2	Mo^a : SDBS = 8 : 1	2^{b}	0.10	25	2.0	submicro-rods
S-3	Mo^a : NaCl=3 : 1	6	0.30°	85	0.6	nanobelts
S-4	Mo^a : $NaNO_3 = 3 : 1$	6	0.30°	85	0.6	Mixtures

 Table 1 Summary of experimental conditions

a. The content of Mo^{6+} in $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$. b. The pH value was adjusted by HNO₃ solution (1.5 mol/L). c. The content of Na⁺. Sodium dodecyl benzene sulfonate (SDBS). Mixtures: submicro-wires, nanobelts, and microflowers.

1.2 Physical characterization

A Philips X' pert MPD instrument using Cu K_{α} irradiation (40 kV) was used to record powder X-ray diffraction (XRD) patterns. FE-SEM images were obtained on a field-emission scanning electron microscope (JSM-6701F, JEOL) operated at an accelerating voltage of 5 kV. The transmission electron microscope (TEM) images and selected area electron diffraction (SAED) were observed in a JEM-1200EX TEM.

1.3 Electrochemical measurements

The ORR activities of $NaNH_4Mo_3O_{10} \cdot H_2O$ were evaluated using a glassy carbon electrode (GCE, diameter 3 mm). To prepare the working electrode, 1 mg of $NaNH_4Mo_3O_{10} \cdot H_2O$ was dispersed in 2 mL of pure ethanol, then 10 µL of the suspension were pipetted on to a GCE. The total $NaNH_4Mo_3O_{10}$ · H_2O loading was 71 μ g cm⁻². A conventional three-electrode was used with a saturated calomel electrode (SCE) as a reference electrode, a platinum wire as a counter electrode, and a glassy carbon electrode as a working electrode. All electrochemical measurements were performed using a CHI660A electrochemical workstation in N, N-dimethylformamide (DMF) contained 0.1 mol/L LiCl, and 1.2 mol/L alcohol at room temperature. A platinum plate electrode $(5 \times 5 \times 0.1 \text{ mm}^3)$ was also used as working electrode for comparison.

2 Results and discussion

The NaNH₄Mo₃O₁₀ \cdot H₂O submicro-wires with the

diameter of 200 ~ 300 nm and the length of about 10 μ m were obtained by reacting (NH₄)₆Mo₇O₂₄ · 4H₂O with SDBS at pH=6 (Fig. 1a). A representative single nanowire and its corresponding SAED pattern are shown in Fig. 1b and the inset respectively, which clearly reveal that the growth direction of this nanowire is along [0 1 0]. As shown in Fig. 1c, the NaNH₄Mo₃O₁₀ · H₂O submicro-rods with the diameter of 200 ~ 300 nm and the length of about 2 μm were obtained at pH = 2. Polyoxomolybdate submicro-wires should grow up in long SDBS micelles at pH=6, but at pH = 2, excessive H^+ cations appeared, and they caused more Mo₃O₁₀²⁻ anions and short SDBS micelles, which leaded to the submicro-rods. Due to the low aspect ratio, the exposure of end faces for submicro-rods is higher than that for nanowires. When $(NH_4)_6 Mo_7 O_{24} \cdot 4H_2 O$ reacts with NaNO₃, the products were the mixture of submicro-wires, nanobelts, and microflowers (Fig. 1d). Because NO₃⁻ could form the H-bonds, which would lower the surface energy of submicro-wires, nanobelts could appear together with submicro-wires and they could self-assemble into microflowers. The pure nanobelts, which could be the stable morphology of NaNH₄Mo₃O₁₀ · H₂O, can be achieved by reacting $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ with NaCl (Fig. 1e), and their growth direction is also along $\begin{bmatrix} 0 & 1 & 0 \end{bmatrix}$ as shown in Fig. 1f and the inset.

As shown in Fig. 2a, all the observed 24 diffrac-



Fig. 1 SEM and TEM images of $NaNH_4Mo_3O_{10} \cdot H_2O$. (a, b) nanowires; (c) nanorods; (d) mixture; (e, f) nanobelts

tion peaks can be perfectly matched to those of orthorhombic NaNH₄Mo₃O₁₀ · H₂O (JCPDS No 36-0335 a =9.25 b=7.58 c=13.47). No peaks of other impurity phases are detected in this pattern, suggesting that the sample should be a nearly pure phase. Moreover, the loss (8.62%) of nanorods that was heated up to 300 °C for 2 h almost equals the calculated loss (8.68%) of NaNH₄Mo₃O₁₀ · H₂O. This also confirms the formation of NaNH₄Mo₃O₁₀ · H₂O. However, the diffraction peaks' intensities of nanobelts and the mixture is stronger than that of nanowires and nanorods, especially that of the (101), (002), (004) and (301) (Fig. 2b), indicating the high degree of crystallinity of nanobelts and the mixture.



Fig. 2 (a) A representative XRD pattern of $NaNH_4Mo_3O_{10} \cdot H_2O$ nanowires; (b) XRD patterns of $NaNH_4Mo_3O_{10} \cdot H_2O$ with different morphologies

In our cyclic voltammetry, two irreversible redox peaks appeared at -0.67 V, -0.89 V, 0.29 V, and -0.12 V in N₂ saturated electrolyte (Fig. 3a), which could be attributed to the two 2-electron reduction process of Mo^{6+[31]} for polyoxomolybdate. For barely GCE, the DMF maybe react with glass carbon and form a film of N-modified carbon, which may catalyze oxygen reduction reaction^[32]. The peak potentials of oxygen reduction in O_2 saturate electrolyte for GCE, submicro-wires, submicro-rods, nanobelts, and mixture were -0.91 V, -0.94 V, -0.88 V, -0.92 V, and -0.93 V respectively. The peak current densities for GCE, submicro-wires, submicro-rods, nanobelts, and mixture were -0.28, -0.27, -0.36, -0.31, and -0.29 mA cm⁻² respectively (Fig. 3b). This result suggested that submicro-rods should be the best electrocatalyst for the ORR among 1D NaNH₄Mo₃O₁₀ · H₂O due to the high exposure of end face. This result may approve that the active sites for ORR could locate on the surface of end face. Though the exposure of end face for nanobelts is low, the high degree of crystallinity may provide enough active crystal plane, which could lead to the excellent activity for ORR. More detailed cyclic voltammograms was given in Fig. 4. Both peak currents increased linearly with the increase of square roots of the scan rates, indicating that the ORR process over polyoxomolybdate nanomaterials was controlled by the diffusion of oxygen to the electrode surface^[12].







Fig. 4 Cyclic voltammetry curves of NaNH₄Mo₃O₁₀ \cdot H₂O nanowires (a), nanorods (b), nanobelts (c), and the mixture (d) in O₂ saturated 0.1 mol/L LiCl in DMF at various scan rates (10, 20, 50, and 100 mV/s). The inset indicates the dependence of oxygen reduction peak current on square root of scan rate

Alcohol can block some active sites for oxygen reduction, which lead to the loss of ORR activity. The effects of different alcohols (methanol, ethanol, and iso-propanol) on the activity of nanorods and nanobelts towards ORR were investigated. As shown in Fig. 5, the peak current densities for GCE, nanorods, and nanobelts increased in the presence of high concentration alcohol in DMF (1.2 mol/L), while the peak current densities of Pt plate electrode decreased greatly in this condition. It indicated that activities of ORR increased when alcohol were added into DMF. The positive effects of alcohol on the enhancement of ORR activity for NaNH₄Mo₃O₁₀ · H₂O nanorods follow the order: ethanol \approx methanol > iso-propanol and that for NaNH₄Mo₃O₁₀ · H₂O nanobelts follow the order: ethanol > methanol > iso-propanol. Alcohol may react with H⁺ in electrolyte, and thus promote the reduction of oxygen. Moreover, it should be noted that, the peak current densities for Pt in DMF (-0.46 mA cm⁻²) is nearly 3 times of that in 0.6 mol/L H₂SO₄ solution (-0.15 mA cm⁻²) (Fig. 5d and e). This suggested that DMF is a better electrolyte than H₂SO₄ solution for



Fig. 5 influence of alcohol (1.2 M) on the activity of GCE (a), nanorods (b), nanobelts (c), and Pt (d) towards ORR in 0.1 mol/L LiCl in DMF: scan rate: 50 mV/s, and Pt in 0.6 M H₂SO₄ solution (e)

high current density. The methanol oxidation potential on Pt in DMF was reduced to 0 V and -0.88 V (Fig. 5d), but that in 0.6 mol/L H₂SO₄ solution was 1.11 and 0.35 V (Fig. 5e). The oxygen reduction potential on Pt also reduced from 0.33 V to -0.91 V. So it is feasible to assemble a fuel cell by using DMF solution as electrolyte.

3 Conclusion

In summary, 1D NaNH₄Mo₃O₁₀ · H₂O, including submicro-wires, submicro-rods, nanobelts and the mixture have been successfully synthesized through a facile process. Owing to the high exposure of end face, submicro-rods exhibited the best activity in ORR in organic solvent among 1D NaNH₄Mo₃O₁₀ · H₂O. The effects of alcohol on the enhancement of ORR activity for submicro-rods may be caused by no oxidation of alcohol, and H⁺ may react with alcohol, and follow the order: ethanol \approx methanol > iso-propanol.

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-维亚微米多钼酸盐形貌可控合成及氧的电催化还原研究

邓忠华,吕功煊*

(中国科学院兰州化物所 羰基合成与选择氧化国家重点实验室,甘肃 兰州 730000)

摘 要:通过调节反应 pH 值和反应物种实现了一维亚微米多钼酸盐的控制合成,以钼酸铵和十二烷基苯磺酸钠 为原料,在 pH=6 时制备出亚微米线,在 pH=2 时制备出亚微米棒,而以钼酸铵和氯化钠为原料,在 pH=6 时制 备出纳米带.在 pH=6 时,纳米材料在长胶束内生长成亚微米线,而 pH 值降低至 2 时,过多的 H⁺不仅会形成大 量的 Mo₃O₁₀⁻²,还会使得胶束变短,因此使得亚微米材料变短,成为亚微米棒.在没有模板剂十二烷基苯磺酸钠 时,多钼酸铵就会长成结构更为稳定的纳米带.循环伏安测试结果表明在有机溶剂中,多钼酸盐的亚微米棒对氧 电催化还原具有优异活性.研究表明,由于有机醇能与电解质溶液中的 H⁺反应,因此有机醇能显著促进一维多钼 酸盐亚微米材料的氧的电催化活性.

关键词:多钼酸盐;一维亚微米结构;氧电催化还原;醇促进作用