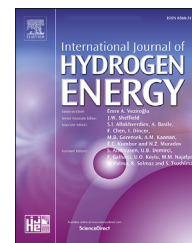




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# In-situ ammonia-modulated silver oxide as efficient oxygen evolution catalyst in neutral organic carboxylate buffer

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## ABSTRACT

Silver-based catalysts with an adjustable structure are promising as efficient oxygen evolution catalysts. Four types of silver-oxide (AgO) morphologies were obtained by potentiostatic deposition in situ in organic carboxylate by altering the amount of ammonia as the complexing agent. AgO films can be used in the oxygen-evolution reaction; AgO that is formed by using potassium acetate that contains 1.32 mM ammonia as an electrolyte exhibits preferable performance. Catalyzed water oxidation occurs in 0.1 mol/L potassium phosphate solution (pH 12.3) with an average activity of 3.5 mA/cm<sup>2</sup>, and persists for at least 10 h at 2.03 V (vs. Reversible Hydrogen Electrode, RHE). Overpotential is 438 mV at a current density of 1 mA/cm<sup>2</sup>. The Faradaic efficiency is 94.4%. Samples were well characterized by X-ray powder diffraction, scanning electron microscopy and X-ray photoelectron spectroscopy.

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## Introduction

Water electrolysis to form hydrogen is an effective way to use solar power, wind power and other intermittent energy [1,2]. Of the two half reactions, the oxygen evolution reaction (OER) is more kinetically demanding because it requires the transfer of four electrons [3,4]. Therefore, the OER is critical to achieve efficient water splitting [1,5–8]. The identification of a more efficient oxygen evolution catalyst (OEC) is a significant challenge.

The research of OECs mainly focused on transition metals. The existing transition metal OECs include cobalt catalysts

[9,10], nickel catalysts [11,12], iron catalysts [13,14], copper catalysts [15,16] and silver catalysts [17–23], among which Ag-B<sub>i</sub> [19,20], Ag-C<sub>i</sub> [21,22] and Ag-P<sub>i</sub> catalysts [23] were prepared in an inorganic salt buffer solution under mild conditions. However, a strong interaction exists between Ag<sup>+</sup> and the anions of almost all inorganic salt buffer solutions, which would not benefit Ag<sup>+</sup> anodic deposition. For example, for the Ag-P<sub>i</sub> catalyst, with a strong force between Ag<sup>+</sup> and PO<sub>4</sub><sup>3-</sup>, the K<sub>sp</sub> for Ag<sub>3</sub>PO<sub>4</sub> is 1.4 × 10<sup>-16</sup>, and a Ag<sub>3</sub>PO<sub>4</sub> colloid forms in the electrolyte solution. Ag<sub>3</sub>PO<sub>4</sub> colloid that adsorbed the excessive negatively charged PO<sub>4</sub><sup>3-</sup> would move to an anode under an applied potential, and be followed by anodic oxidation and electron transfer. In light of the in-situ formation, a dynamic

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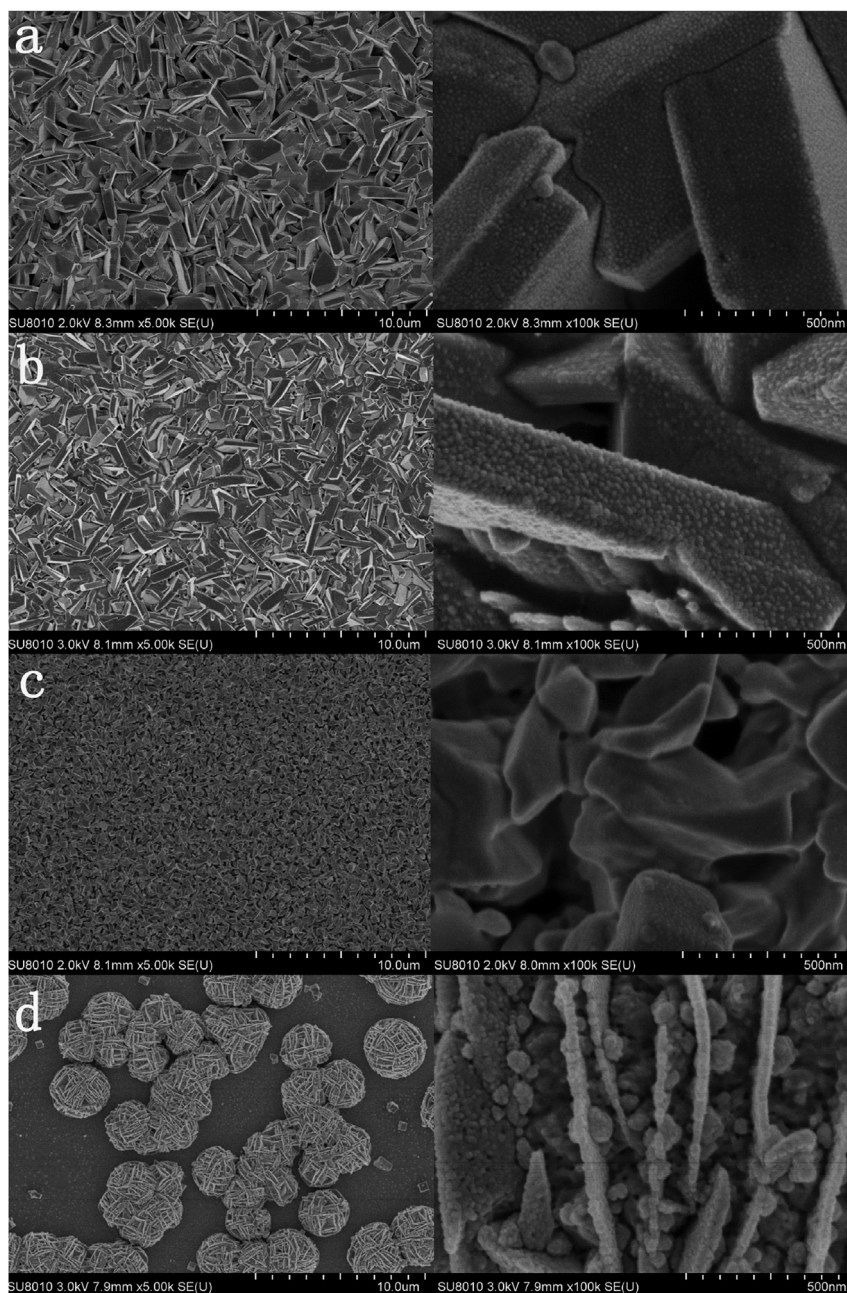
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**Table 1 – Experimental conditions used for the synthesis of Ag-based catalysts with different concentrations of ammonia (1) 1.32 mM, (2) 2.31 mM, (3) 4.95 mM, (4) 6.27 mM.**

Sample	1	2	3	4
$C_{\text{NH}_3\text{H}_2\text{O}}/\text{mM}$	1.32	2.31	4.95	6.27

equilibrium may exist between  $\text{Ag-PO}_4^{3-}$  in solution and  $\text{Ag}^+/\text{Ag}^{2+}-\text{PO}_4^{3-}$  on the electrode. However, because of the large amount of  $\text{PO}_4^{3-}$  in the electrolyte solution, a tiny change in  $\text{Ag}^+$  concentration influences this equilibrium significantly, which is not conducive to the deposition of  $\text{Ag}^+$ , and could impact its catalytic performance.

Organic carboxylic acid salt was selected as a buffer electrolyte; however, an Ag-based catalyst could hardly be obtained with potassium acetate as the electrolyte on the ITO electrode by electrodeposition. The pH of the 0.1 M potassium acetate (pH 7.5) is lower, and is under nearly neutral pH conditions, which has a negative effect on  $\text{Ag}^+$  anodic electrodeposition. If KOH is added to adjust the pH of  $\text{CH}_3\text{COOK}$ , it would have increased the bonding interaction between  $\text{Ag}^+$  and  $\text{OH}^-$ . The formation of  $[\text{Ag}(\text{NH}_3)_2]^+$  species enables the growth of Ag-based nanocrystals with a high degree of control [24]. Organic carboxylic acid salt with  $\text{NH}_3 \cdot \text{H}_2\text{O}$  was used as a buffer electrolyte to avoid  $\text{AgOH}$  precipitation ( $K_{\text{sp}} = 2 \times 10^{-8}$ ) and to change the solution pH. The formation of an  $[\text{Ag}(\text{NH}_3)_2]^+$  complex is critical for the successful fabrication of



**Fig. 1 – SEM images and corresponding high-magnification SEM images of samples #1–4 at 2.03 V (vs. RHE) from different concentrations of  $\text{NH}_3 \cdot \text{H}_2\text{O}$ : (a) 1.32 mM, (b) 2.31 mM, (c) 4.95 mM, (d) 6.27 mM.**

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