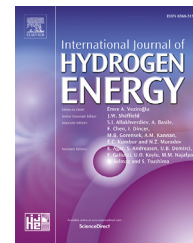




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Boosting the electrocatalytic performance of Pt, Pd and Au embedded within mesoporous cobalt oxide for oxygen evolution reaction

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ABSTRACT

High efficient and low-cost electrocatalysts for the oxygen evolution reaction (OER) are essential components of renewable energy technologies. However, the high onset potential and limited active sites restrict the catalytic activity of current electrocatalysts. Therefore, we are motivated by the development of cheap and efficient catalytic electrodes to promote the sluggish water-splitting systems associated with the large-scale application of clean and renewable energy technologies. Herein, a novel, simple, and efficient routine is presented by noble metal particles embedded within mesoporous metal oxide materials as high-efficiency anode catalysts for OER. Highly ordered mesoporous Co₃O₄ was prepared by a nanocasting method using the silica KIT-6 as hard template, showing an enhanced electrochemical performance. Then, M - Co₃O₄ (M = Pt, Pd, Au) nanomaterials were prepared by a simple but novel chemical reduction method. They show the high surface area of 112.3, 81.0 and 73.6 m² g⁻¹, which can provide more active surface area exposure leads to shorter paths of charges from electrolyte to electrode surface. Moreover, a three-dimensional highly ordered mesoporous structure can facilitate diffusion and penetration of electrolyte and oxygen, and can also keep catalyst nanoparticles in a well-dispersed condition with more active sites. Electrochemical measurements revealed that 20, 50, 25 wt % are the best weight contents for M (M = Pt, Pd, Au) in the Co₃O₄ with highest electrochemical activity (0.410, 0.415 and 0.422 V vs. SCE) and j_{0.7V} reaching a maximum value. M-Co₃O₄ (M = Pt, Pd, Au) materials exhibit superior activities and excellent long-duration stability in alkaline attributed to accelerating the formation of Co(IV) cations after being introduced M (M = Pt, Pd, Au) nanoparticles within mesoporous Co₃O₄. This kind of noble metal embedded within mesoporous oxide catalysts will hold a large potential as a highly promising electrocatalyst in the future.

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Introduction

The increasing demand for energy and diminishing natural energy resources have led to a revolutionary era of discovering earth-abundant energy alternatives and designing efficient energy–storage devices [1,2]. Currently H_2 production still mostly relies on fossil fuel industry and suffers from low purity [1,3,4]. One of the most efficient ways of producing H_2 with high purity is water splitting into hydrogen and oxygen by electrolysis [5,6]. The oxygen evolution reaction (OER), as an important half-reaction involved in water splitting, has been intensely investigated for decades [7–11]. OER is a demanding step that includes four proton-coupled electron transfer and oxygen-oxygen bond formation, so it is not kinetically favored and requires large overpotential [12]. Large-scale electrolysis of water is greatly hindered by sluggish anodic OER [13]. Therefore, an appropriate OER catalyst is essential to accelerate the reaction rate and decrease the overpotential. Iridium dioxide (IrO_2) and Ruthenium dioxide (RuO_2) are OER electrocatalysts with low overpotential, especially in acidic conditions [14,15]. However, these catalysts suffer from scarcity and high cost, and cannot be utilized in large-scale water splitting industries to obtain economic H_2 energy resources. Therefore, extensive research effort has been devoted to developing highly active, durable and low-cost OER electrocatalysts. Recently, transition metal oxides, especially Co oxide nanocomposites which exhibit comparable or even higher activities than IrO_2 or RuO_2 have drawn more and more attention [16–19].

Meanwhile, nanostructuring a material is a more effective method for increasing the active surface area per mass, because the more active surface area exposure leads to shorter paths of charges from electrolyte to electrode surface, higher specific surface area and quantum confinement effects directly. Previous studies have shown that electrocatalysts for OER are usually in form of agglomerated sphere-like particles, agglomerate particles, nanocluster or nanotubes [10–12,15,16,18,20]. For such structures, electrocatalytic activity may significantly limited by small surface area and poor electron–mass transfer. To address this situation, various morphologies of mesoporous materials for OER process have been studied widely such as nano-structured needle-shaped [14], porous nanowires [19], nanowire arrays [21], hierarchical nanorod [22], mesoporous nanoplatelet [23] and so on. Zou and co-workers reported a facial synthetic method to porous Ni-doped Co_3O_4 nanostructure materials in order to against aggregation/agglomeration and this 3D (three-dimensional) nano-network structure exhibits enhanced catalytic activity for OER [24]. Specifically, ordered mesoporous cobalt oxide with non-spherical shapes and regular interiors have become more attractive because of their designable morphology, low density, large void space, high surface area and shell permeability [25]. This kinds of materials can be prepared via hard templating and soft templating (cooperative assembly) pathways. However, after removal of surfactant template, metal oxides are prepared by soft templating exhibit low thermal stability. Therefore, nanocasting method which uses mesoporous silica, such as KIT-6 and SBA-15 as hard templates has emerged as a general strategy. A wide range of ordered

mesoporous morphologies have been synthesized in the past decade using these hard template, such as $NiO-CuO$ [26], $NiMoO_4$ [27], $NiCo_2O_4$ [28], $LaMO_3$ ($M = Mn, Fe$) [29], $Fe_2O_3-ZrO_2$ [30].

Recently, it has been illustrated that formation of $Co(IV)$ cations is a crucial process to OER [31]. $Co(IV)$ cations are involved as intermediate states or mediator sites, which will accelerate the generation of oxygen at the reaction interphase in the OER. Therefore, the addition of other active materials into Co_3O_4 which could increase the population of active centers and promote the formation of $Co(IV)$ on the catalyst surface is of great importance. Among them, modifying with highly electronegative metals, such as Pt [32], Pd [33] and Au [34] has been demonstrated as some most effective methods to improve the electrocatalytic efficiency. For further improvement the OER catalytic properties, recent reports have shown that oxides modified by noble metals such as Pt- $CaMnO_3$, Pd- Mn_3O_4/C , Pd- Co_3O_4 , Au- CoO_x , M/MnO_x ($M = Pt, Pd, Au, Ag$), M- NiO/C ($M = Pt, Pd, Au$) can enhance the catalytic activity for OER [32–39].

Recently, Yeo and et. reported that the higher OER can occur on cobalt oxide films deposited on Au and other metal substrates. But this method requires the use of bulk Au as supporting substrate for Co_3O_4 which lacks of porous structure, therefore the limited amount of Co may restrict the performance at high current densities [35]. Lu and co-workers studied the Au nanoparticles embedded within mesoporous cobalt oxide enhancing the electrochemical OER. But the synthesis of Au/ mCo_3O_4 composites is cobalt precursors mixing directly with gold chloride trihydrate solution, which may cause the inhomogeneous distribution of this two electrocatalyst active sites, therefore restricted the electrochemical performances [36]. Our group before also explore the different noble metal particles well dispersing on NiO/C , benefiting the synergistic effect, M- NiO/C ($M = Pt, Pd, Au$) exhibits superior electrochemical activity and excellent stability for OER. But because of some aggregation structure of the oxide shown in the SEM results, it may influence the contact homogeneity between the integral catalysts and electrolyte, subsequently impact the electrochemical performance [38]. Therefore, to address above problems, uniform and ordered mesoporous structure is introduced to be the substrate or skeleton of the electrocatalysts by chemical reduction method, which can present a homogeneous and highly ordered morphology of both the cobalt oxide and Pt, Pd and Au nanoparticles embedded in. The 3D mesoporous Co_3O_4 and M- Co_3O_4 ($M = Pt, Pd, Au$) will be applied as electrocatalysts for the OER. After testing the potential and current density of the electrocatalysts in different ratio between cobalt oxide and noble metal respectively, the values of the best weight contents for M ($M = Pt, Pd, Au$) in the M- Co_3O_4 ($M = Pt, Pd, Au$) materials with the lowest onset potential and highest current density are also be studied and tested their performance repeatedly. Meanwhile, we compare the electrochemical performances between these three noble metal embedded within electrocatalysts in optimal proportion with common Co_3O_4/C material. These pioneering studies have inspired us to combine the advantages of both metal oxides and noble metal particles, and to design different highly order mesoporous structures of nanocomposites for highly efficient oxygen evolution reactions.

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