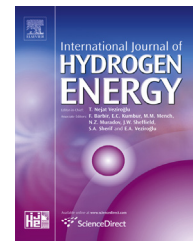




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Maghemite nanorods anchored on a 3D nitrogen-doped carbon nanotubes substrate as scalable direct electrode for water oxidation

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ABSTRACT

A hybrid catalyst 3D electrode for electrochemical water oxidation to molecular oxygen is presented. The electrode comprises needle shaped maghemite nanorods firmly anchored to nitrogen doped carbon nanotubes, which in turn are grown on a conducting carbon paper that acts as efficient current collector. In 0.1 M KOH this hybrid electrode reaches a current density of 1 mA/cm² (geometric surface) at an overpotential of 362 mV performing high chronoamperometric stability. The electrochemical attributes point toward efficient catalytic processes at the surface of the maghemite nanorods, and demonstrate a very high surface area of the 3D electrode, as well as a firm anchoring of each active component enabling an efficient charge transport from the surface of the maghemite rods to the carbon paper current collector. The latter property also explains the good stability of our hybrid electrode compared to transition metal oxides deposited on conducting support such as fluorine doped tin oxide. These results introduce maghemite as efficient, stable and earth abundant oxygen evolution reaction catalyst, and provide insight into key issues for obtaining practical electrodes for oxygen evolution reaction, which are compatible with large scale production processes.

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Introduction

Oxygen evolution through (photo) electrochemical oxidation of water is a key reaction in energy conversion processes taking place in metal–air batteries [1,2] and artificial photosynthesis devices [3–5]. In nature, photosynthesis utilizes solar energy for converting water and carbon dioxide into

energy-rich carbohydrates and molecular oxygen. In artificial photosynthesis, the aim is to mimic the efficient primary reactions of photosynthesis for the direct conversion of the abundant solar light and water into molecular oxygen and a storable fuel such as molecular hydrogen or low chain alcohols. Since burning hydrogen in combustion engines or fuel cells enables the production of work or electrical energy with

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only water as a byproduct, artificial photosynthesis is a highly attractive route for the production of renewable fuels [6].

Although some materials are able to photocatalytically oxidize water by the influence of ultraviolet light, this process is problematic for industrial use due to the mismatch with the solar spectrum and serious issues of charge recombination [4,7]. A more technologically interesting solution relies on the concept of an artificial leaf comprising a photovoltaic (PV) cell that can drive two catalytic processes, one which oxidizes water to oxygen and protons (referred to as either water oxidation reaction, WOR, or oxygen evolution reaction, OER), and one which reduces the protons to hydrogen (hydrogen evolution reaction, HER). Two main techniques can be distinguished for such devices. In direct solar-to-fuel conversion, the catalyst electrodes are in direct contact with the PV cell [8–10], while in wired devices the catalyst reactions are usually separated from those occurring in the PV cell [11–13]. The former concept of wireless, “direct-contact” photo electrochemical cells (artificial leaf) has the potential for higher efficiency and lower system costs, but is more prone to stability issues due to the direct contact of the photoactive layer with water. The concept has been proven to work and to generate solar-to-fuel efficiencies (SFE) as high as 4.7% [8,10]. However, the possibility to separately tune the PV cell and the electrocatalysts [10] has resulted recently in significantly higher SFE for wired devices. Utilizing either four silicon PV cells connected in series [14] or a single perovskite PV cell [11], SFEs as high as 10.2% and 12.3% have been demonstrated. The proof of concept presented by Cox et al. [14] and Luo et al. [11] elegantly shows that appropriate electrocatalyst electrodes can be wired to the PV cell to obtain an efficient solar-to-energy device. To take full advantage of this concept, the development of efficient electrocatalyst based on earth-abundant elements that can be easily mass produced and up-scaled is required.

Here we describe a route to develop a direct OER electrode for which the electrocatalyst is directly grown on the surface

of the current collector. Our work is based on three guiding principles; i) Avoid the use of scarce and expensive noble metal based oxides, such as IrO_2 and RuO_2 , despite their documented efficiency [15–17]. This allows up-scaling and reduces costs. ii) Establish an electrode which maximizes the electrocatalytic area, but concurrently minimizes the overall resistance from the reaction site to the current collector. This is motivated by the observation that the OER activity and turnover frequency of a ~ 0.4 monolayer of cobalt oxide deposited on electrochemically roughened Au is reported to be forty times higher than that of bulk cobalt oxide [18]. iii) Synthesize the electrocatalyst in a bottom-up process that directly integrates the electrocatalyst on the electrode, and which is compatible with up-scaled production techniques. This increases electric conductivity and stability.

Considering these design principles we developed a hybrid material comprising of maghemite ($\gamma\text{-Fe}_2\text{O}_3$) nano-sized rods anchored on nitrogen-doped carbon nanotubes (NCNTs), which in turn are grown on a conductive carbon paper (CP) serving as a current collector (see Fig. 1). Even though, first-row transition metal oxides and iron-based electrocatalyst have been well studied before [19–23], to our knowledge, this is the first report of maghemite as an electrocatalyst for OER. Also, the combination of maghemite nanorods with NCNTs, which not only work as an electron-conducting material but also provide strong attachment sites, results in a 3D electrode with very good electrochemical water oxidation activity. In earlier studies we have reported that the strategy of combining NCNTs and CP as metal oxide catalyst support for different catalytic reactions leads to hybrid materials with superior performance [24,25]. In 0.1 M KOH, an overpotential of 346 mV is needed for OER, which is comparable to other state-of-the-art OER electrocatalysts [9,20,26–28], and photoelectrocatalysts [29–32], that are based on earth-abundant metals. Our 3D electrodes have attractive features, such as an easy preparation method, the possibility to scale up the process, and good stability. In addition our method

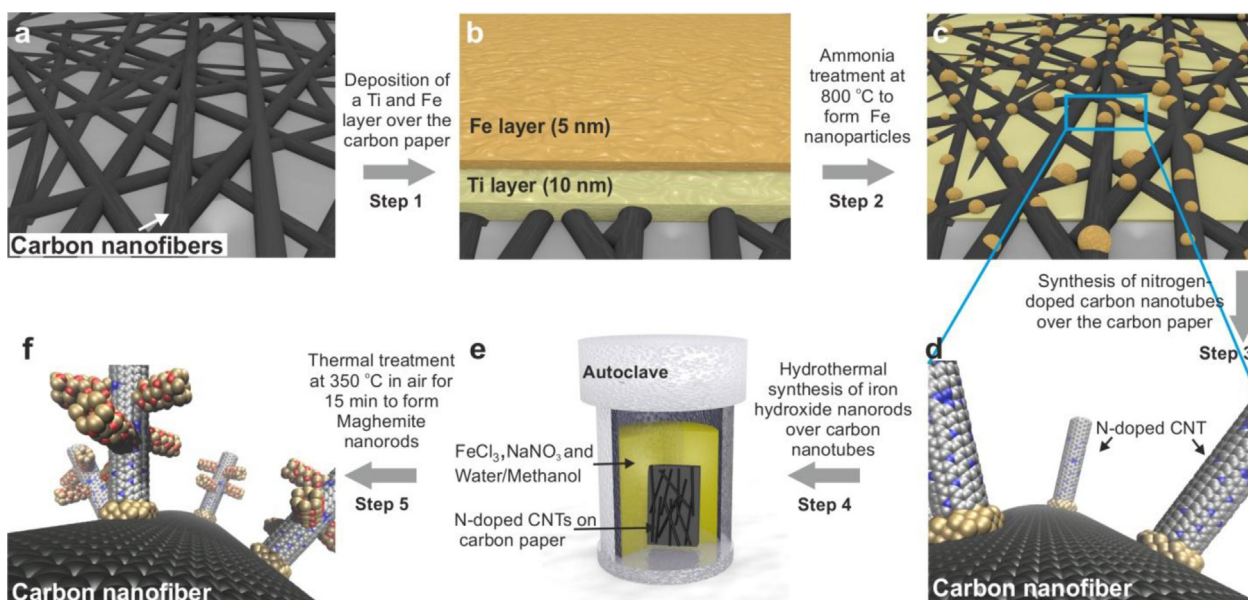


Fig. 1 – Schematic picture of the synthesis route to prepare the hierarchical MR@NCNTs/CP structures (a–f).

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