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Electro-catalyst Formulation from Terbium Self-Supported to Split Water

Al-Baraa Akram*

Affiliation: Faculty of Health and Life Sciences, Department of Pharmaceutical Biotechnology, Portland Laboratory in De Montfort University in Leicester, United Kingdom

***Corresponding author:** Al-Baraa Akram, Faculty of Health and Life Sciences, Department of Pharmaceutical Biotechnology, Portland Laboratory in De Montfort University in Leicester, United Kingdom, E-mail: albraaakram94@gmail.com

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Abstract

A wide range of applications require the design of excellent, affordable, and stable electro-catalyst systems for practical applications. One promising method and tactic for creating a sustainable and clean energy source is large-scale electrolytic water splitting. In this work, we use a hydrothermal technique to stepwise surface-redox-etch Tb Foam (TF) to create a self-supported electro-catalyst from Tb-CoB LDH-Tb-CoO electro-catalyst. The Tb-CoB LDH-Tb-CoO was produced. Water oxidation produces a current with a density of roughly $100\text{mA}\cdot\text{cm}^{-2}$ at a voltage of just 240 mV. The hydrogen evolution reaction, on the other hand, requires 276 mV in 1 M potassium hydroxide. In a two-electrode alkaline electro-catalyst, the synergistic impact of Tb-CoB LDH-Tb-CoO results in the development of a highly self-supported catalytic system that can split water by crossing a $10\text{mA}\cdot\text{cm}^{-2}$ current density at just 1.53 V. To ensure durability and potential as a real-world energy conversion system, the developed electrode generates a stable performance for an extended period of time with a high density of current.

Keywords: Terbium, Electro-catalyst, Layer Double Hydroxide.

Abbreviations: LDH- Layered Double Hydroxides, TF-Terbium Foam, CC-Carbon Cloth, SCE-Saturated Calomel Electrode.

Introduction

The over-growing need and demand of fossil fuel direct pushed modern technology to discover and invent new, clean and sustainable energy sources to be applicable widely in our life. Water splitting is one of these attempts to get cheap energy source from hydrogen as water is available and abundant in most Earth area which is about 70%. This technology is promising to produce hydrogen molecules H_2 which has a great potential to meet the current global energy demands due to the very high density of the current energy extracted from the current fuel sources. Water splitting consists of two half-cell reactions which are oxygen evolution reaction and hydrogen evolution reaction. Oxygen evolution reaction represents the anodic reaction which is uphill thermodynamically and requires a high over-potential [1]. The benchmark catalysts for high performance of oxygen evolution reaction and hydrogen evolution reaction generally include novel metal based materials as ruthenium and iridium to represent oxygen evolution reaction, and platinum for example to represent hydrogen evolution reaction. These metals should resist their large scale application due to high cost and rare existence. So, the fabrication of cost effective metal based materials is highly desirable to replace expensive catalysts.

According to this regard, several alternatives including lanthanides and transition metals with their oxides are widely used, hydroxides, sulfides, selenides, phosphides and nitrides in both mono and multi-metallic forms are investigated for their efficient properties in water splitting [2-5].

Recently, Layered Double Hydroxides (LDH) have emerged as a class of promising oxygen evolution reaction catalysts in alkaline media. Although the intriguing morphologies, amazing crystal structures and

superficial anion exchange capability of LDH favor electrochemical performance, their poor intrinsic conductivity always affect the overall performance [6]. So, maximum exposure to the surface active sites, high charge/mass transport properties and easy release of gas facility are to be put into considerations before designing the electro-catalysts [7,8]. Nickel-iron Layered Double Hydroxides and gadolinium-cobalt were previously reported by various papers, where the fascinating oxygen evolution reaction performance was explained by the increase in catalytic surface area and high effect by the incorporation of metals [8,9].

However, Ni-Fe LDH as an overall water splitting catalyst is not very effective, which inspires scientists for its coupling with other nano-components to fabricate interface engineered hybrid nano-materials for whole cell water splitting. In contrast, Tb-CoO is explored as hydrogen evolution reaction catalysts or even bifunctional electro-catalysts due to their inherent activity and long term stability. Moreover, in a two dimension planar structure the number of active sites are near from the atoms in the outside surface of the atom [10,11].

Motivated by these promising approaches, I have designed an electro-catalyst on conductive terbium foam composed of thin layered Tb-CoB LDH and Tb-CoO. This idea shows a massive improvement in performance of water splitting compared to that of the previously reported Ni-Fe LDH and nickel selenide/nickel foam based hybrid materials [8]. The designed ternary electro-catalyst Tb-CoB LDH-Tb-CoO/TF demonstrates efficient water splitting in a system of two electrodes with a low cell voltage reaches 1.63 V for $10\text{mA}\cdot\text{cm}^{-2}$ current density in an alkaline medium with very long term stability.

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Currently, highly active electro-catalysts are constructed concentrating on high surface area, remarkable conductivity and outstanding exposure of the active sites for the sake of increasing their performance toward hydrogen evolution reaction and oxygen evolution reaction. For this purpose, catalysts with different morphologies, such as hollow/porous, hierarchical architectures are often found to be integrated with the conductive networks such as any metal foam, carbon cloth and so forth [4,12].

Benefitting from the synthetic strategy, the self-supported catalysts are found to be cost effective and mechanically more stable compared to the fabricated electrode with an extra insulating polymeric layer as a binder like nafion or polytetrafluoroethylene. Moreover, a single material with perfect catalytic activity for both hydrogen evolution reaction and oxygen evolution reaction are of special interest to achieve the water splitting more feasibly. It should be mentioned here that the electrolytes suitable for hydrogen evolution reaction or acidic medium and oxygen evolution reaction or alkaline medium for the non-novel metal based electro-catalysts are always mismatched with each other. However, in applicable life cases both of the half-cell reactions demand to function in the same electrolyte to participate in making the overall water splitting straightforward with the least loss of energy.

Materials and Methods

All the reagents are used without any purification. Cobalt chloride hexahydrate $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, boric acid H_3BO_3 , Terbium Foam (TF), Carbon Cloth (CC), ethanol, hydrochloric acid, potassium hydroxide (KOH) and deionized water which is used during the process of the investigation.

Tb-CoB LDH-Tb-CoO/TF synthesis

It is synthesized by using Tb-CoO/TF cleaned with acetone and pretreated with 3M hydrochloric acid for 15 minutes to get rid of the surface oxides under sonication condition. Terbium foam is used as a substrate with 0.3mmol of Co^{+3} salt for the deposition of Tb-CoB LDH. The calculated average loading mass is 2.06mg cm^{-2} approximately.

Preparation of electrodes from the commercial catalysts

10.12 mg of 20% Pt-C and 2.07 mg RuO_2 are dispersed separately in $10\mu\text{L}$ 5% nafion and 990 μL mixture of ethanol and water by ratio 1:1 by ultra-sonication for one hour. Then pretreated terbium foam is coated with these substances so the loading mass should reach 2 mg cm^{-2} approximately. Then it should be dried under ambient conditions.

Electrochemical study

Oxygen evolution reaction and hydrogen evolution reaction are performed in a three electrode glass cell at ambient temperature using a Saturated Calomel Electrode (SCE) as a reference, platinum disc electrode used as a counter electrode, and the prepared terbium foam based materials are used as working electrodes. The two-electrode water splitting tests were carried out using a glass cell and prepared materials are used as both cathode and anode in 1 M potassium hydroxide solution.

Potentials are referenced to a reversible hydrogen electrode according to this equation:

$$(E_{\text{RHE}} = E_{\text{SCE}} + 0.25 + 0.059\text{pH})/\text{V}$$

Nitrogen adsorption-desorption measurements were performed using a micrometric system after the samples were degassed completely in a vacuum overnight. Our synthetic process differs from other common protocol as terbium ions are not introduced from outside rather than the lost terbium ions from terbium foam which are forming Tb-CoB LDH-Tb-CoO. So, the weight increment per area of terbium foam is not related directly to loading mass of the catalyst on terbium foam.

However, chemical composition of the prepared catalyst will help to estimate the catalyst loading according to the equation below:

$$\text{Mass loading} = x \text{ mg cm}^{-2} * (\text{My}/\text{Mz})$$

While My represents the total molecular weight of the catalyst while Mz represents its terbium free molecular weight. Here we have reported the average mass loading on terbium foam for three synthetic operations (Table 1).

Redox couple	Standard reduction potential (E^0/V) vs. SHE at 25°C
Tb^{3+}/Tb	+3.1
$\text{H}_3\text{BO}_3/\text{B}$	-0.8698
$\text{Co}^{3+}/\text{Co}^{2+}$	+2.8

Table 1: Standard reduction potential for different redox couples.

X-ray diffraction of the material was recorded on X-ray diffractometer. Field emission scanning electronic microscope was used to examine the morphology of the prepared materials whereas images with transmission electron microscope were recorded after sonication of terbium foam based material in ethanol for half an hour. Then the dispersed material was drop casted on carbon coated copper grid. The elemental analyses were recorded using transmission electron microscope. X-ray photoelectron spectrometer was performed using monochromatic laser beam for the analysis of the composition and valence the state of elements present in the synthesized compounds (Figures 1 and 2).

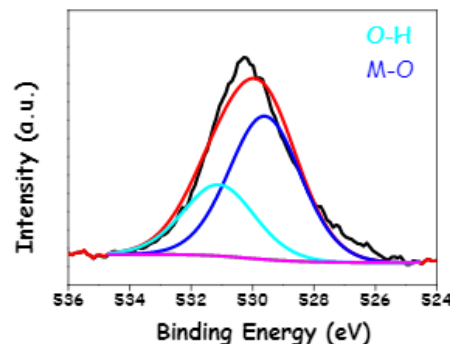


Figure 1: X-ray photoelectron spectrometer for Tb-CoB LDH-Tb-CoO showing intensity versus binding energy by electron volt.

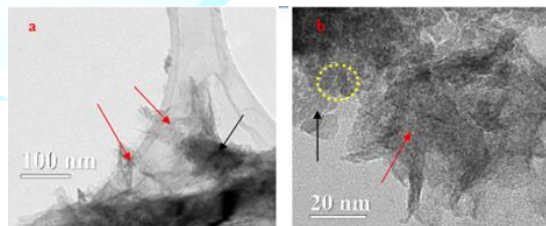


Figure 2: Images taken by transmission electron microscope for Tb-CoB LDH-Tb-CoO.

Results

In Tb-CoB LDH-Tb-CoO, first Tb-CoO is formed on terbium foam surface then Tb-CoB LDH plates like nanostructures are deposited on them. These two parts are found to be separated in transmission electron microscope images which may come from the strong sonication of the sample before transmission electron microscope analysis. However, their close relation can be found in various positions in the transmission electron microscope analysis. So, a strong interaction between the nanoplates of Tb-CoB LDHs and Tb-CoO

nano-sheets is clearly evidenced in figure 2 and this physical connection is helpful for remarkable long-term stability even at higher current density (Figure 3).

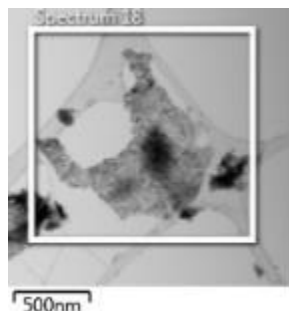


Figure 3: Energy dispersive spectroscopy of Tb-CoB LDH-Tb-CoO.

Terbium is more preferable than other metals and is considered as a better option in manufacturing of electro-catalysts to mix with f orbital as other lanthanides as gadolinium has electrons in d and f orbitals (Table 2) (Figures 4 and 5).

Element	Line Type	K factor	Absorption correction	Wt %	Wt% sigma	Atomic%
Co	K series	1.047	1	18.70%	0.84	21.48%
Tb	K series	0.996	1	44.45%	1.09	48.57%
B	K series	1.123	1	36.85%	1.05	29.94%
Total				100%		100%

Table 2: Line type, k factor, absorption correction and atomic percentage of cobalt, terbium and boron.

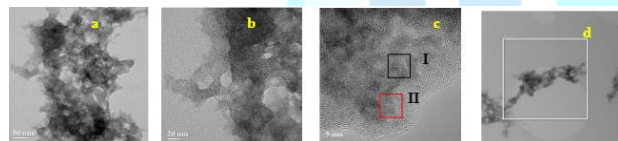


Figure 4: Transmission electron microscope spectra for Tb-CoO/terbium foam.

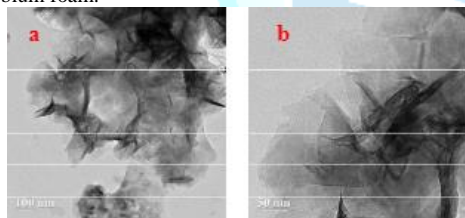


Figure 5: Transmission electron microscope for Tb-CoB LDH/terbium foam.

The designed Tb-CoB LDH-Tb-CoO catalyst produces a stable current with a density of 10 mA cm⁻² for 75 hours. At high current density more than 100 mA cm⁻², the material shows almost 85% activity retention for 75 hours of electro-chemical test. The commercial catalyst couple on terbium foam fails to exhibit stability for only 25 hours (Figure 6).

By testing the performance of water splitting from ternary materials with different amounts of cobalt, Tb-CoB LDH-Tb-CoO/TF-1 and 2 were fabricated from Tb-CoO/TF reacting with 0.1 and 0.3 mmol CoCl₂ respectively. According to this, the cobalt content in Tb-CoB LDH-Tb-CoO which is prepared from 2 mmol of cobalt chloride is optimum for its outstanding water splitting performance (Table 3) (Figures 7 and 8).

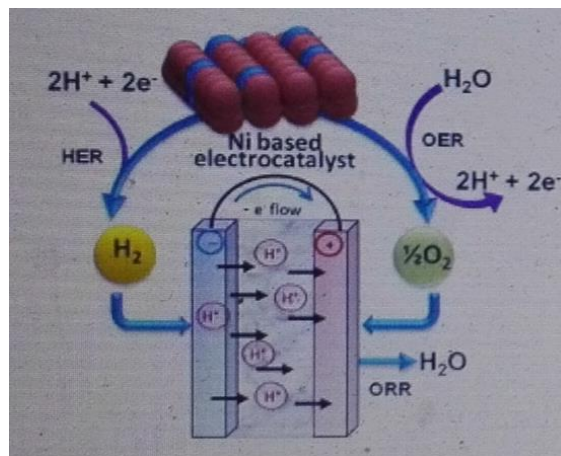


Figure 6: Scheme showing the mechanism of water splitting using a nickel based electro-catalyst. Oxygen evolution reaction on the right represents anodic reaction and hydrogen evolution reaction on the left represents cathodic reaction.

Materials	Oxygen Evolution Reaction (OER)	Hydrogen Evolution Reaction (HER)	Cell voltage for overall water splitting (V) at J=10mA cm ⁻²
Ni ₈ P ₃ /NF	η ₃₀ =270mV	η ₁₀ =130mV	1.61
Ni-P/NF	η ₁₉₁ =350mV	η ₁₀ =150mV	1.64
NiFe-LDH/NiCo ₂ O ₄ /NF	η ₅₀ =290mV	η ₁₀ =192mV	1.6
Ni ₃ S ₂ /NF	η ₁₀ =260mV	η ₁₀ =223mV	1.76 for 13 mA cm ⁻²
NiCoP/NF	η ₅₀ =280mV	η ₁₀ =32mV	1.58
NiFe/NiCo ₂ O ₄ /NF	η ₁₂₀₀ =340mV	η ₁₂₀ =140mV	1.67
NiSe/NF	η ₁₀₀ =314mV	η ₁₀ =96mV	1.63
Co-Ni-Se/C/NF	η ₅₀ =300mV	η ₁₀₀ =183mV	1.6
Ni ₃ Se ₂ nanoforest/NF	η ₁₀₀ =353mV	η ₁₀₀ =279mV	1.61
NiFe-LDH/NF	η ₁₀ =240mV	η ₁₀ =210mV	1.7
CoFe-LDH-F/NF	η ₁₀₀ =310mV	η ₁₀₀ =255mV	1.63
FeNi ₃ N/NF	η ₁₀ =202mV	η ₁₀ =75mV	1.62
Ni(OH) ₂ /NF	η ₁₀₀ =400mV	η ₁₀₀ =303mV	1.68
EG/Co _{0.85} Se/NiFe-LDH	η ₂₇₀ =290mV	η ₁₀ =260mV	1.67
NiS/NF	η ₅₀ =335mV	η ₂₀ =158mV	1.64
NiFe-LDH-NiSe/NF	η ₁₀₀ =240mV	η ₁₀₀ =276mV	1.53
Tb-CoB LDH-Tb-CoO	η ₁₀₀ =324mV	η ₁₀₀ =213mV	1.63

Table 3: Comparison of the water splitting performance of the ternary electro-catalysts.

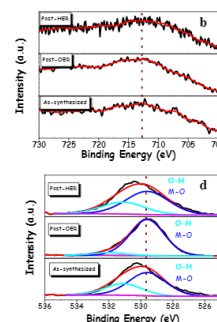


Figure 7: Comparative spectra for Co and O for Tb-CoB LDH-Tb-CoO before and after Hydrogen evolution reaction/Oxygen evolution reaction study.

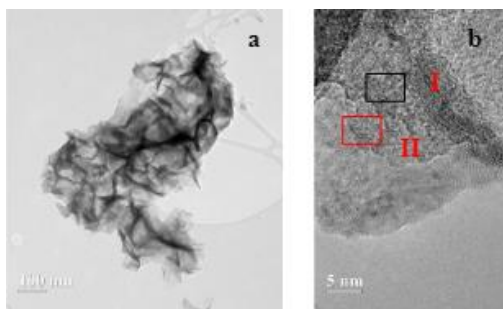
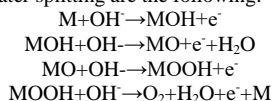


Figure 8: Transmission electron microscopic analysis for Tb-CoB LDH-Tb-CoO /terbium foam after oxygen evolution reaction. I represents 0.23nm Tb-CoB LDH and II represents 0.27 nm Tb-CoO.

The equations of water splitting are the following:



These equations are easily explained in the following scheme (Figures 9 and 10) (Tables 4 and 5).

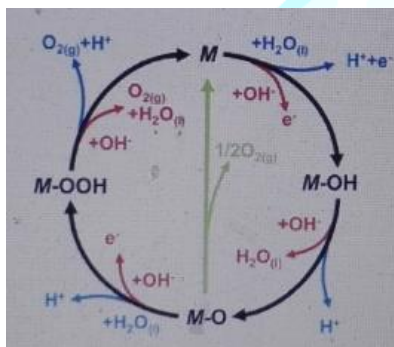


Figure 9: Scheme explains the equations of water splitting where M represents the electro-catalyst. Black lines represent the basic medium reactions and the red lines represent the acidic medium reactions.

Element	Line type	Absorption correction	Wt%	Wt% sigma	Atomic %
B	K series	1	46.89%	0.51	76.71%
Co	K series	1	3.14%	0.17	1.47%
Tb	K series	1	46%	0.48	20.5%
O	K series	1	3.97%	0.19	1.32%
Total			100%		100%

Table 4: Line type, k factor, absorption correction and atomic percentage of cobalt, terbium, oxygen and boron after oxygen evolution reaction.

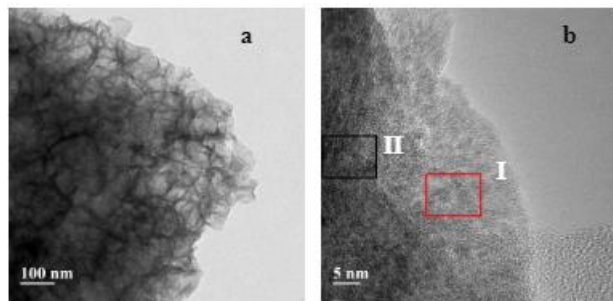


Figure 10: Transmission electron microscopic analysis for Tb-CoB LDH-Tb-CoO /terbium foam after hydrogen evolution reaction. II represents 0.23nm Tb-CoB LDH and I represents 0.27 nm Tb-CoO.

Element	Line type	Absorption correction	Wt%	Wt% sigma	Atomic %
B	K series	1	43.67%	0.46	74.03%
Co	K series	1	19.12%	0.29	9.29%
Tb	K series	1	32.98%	0.37	15.23%
O	K series	1	4.23%	0.16	1.45%
Total			100%		100%

Table 5: Line type, k factor, absorption correction and atomic percentage of cobalt, terbium, oxygen and boron after hydrogen evolution reaction.

Discussion

Designing a self-supported water splitting catalyst for simultaneously boosting both hydrogen evolution reaction and oxygen evolution reaction is highly desirable, which is decisive for enhancing the efficiency of the whole process of water splitting. Inspiration by the proper hydrogen atoms binding energy of terbium and cobalt metal, favorable oxygen evolution reaction kinetics of terbium foam and cobalt chloride, and more efficient electron transfer of self-supported structure, Tb-CoB LDH-Tb-CoO /terbium foam coated in carbon cloth is synthesized by a facile self-assembly carbonization method which is remarkably exhibits much more increased electrochemical activities towards both oxygen evolution reaction and hydrogen evolution reaction with low over-potentials which its Cell voltage for overall water splitting (V) reaches 1.63 V which is required to achieve the current density of 10 mA cm^{-2} .

This is superior to most of the reported CoO and any Co-based catalysts. The most important fact that the fabricated Tb-CoB LDH-Tb-CoO /terbium foam electro-catalyst only needs a small cell voltage between 1.63 V and 1.66 V for driving the whole water splitting to reach the current density of 10 mA cm^{-2} . Moreover, the Tb-CoB LDH-Tb-CoO /terbium foam electro-catalyst has a superior stability after 25 hours of continuous operation. Excellent features including synergistic effect between cobalt and CoO are considered as unique porous structure and self-supported construction impart jointly Tb-CoB LDH-Tb-CoO /terbium foam electro-catalyst which has highly elevated multi-functional electro-catalytic activities (Figure 11).

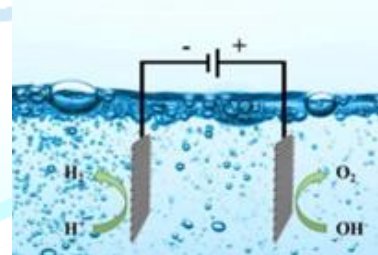


Figure 11: Diagram showing how the electro-catalyst works for splitting water. It contains two electrodes with two side layers from Tb-CoB LDH-Tb-CoO /terbium foam.

Low cost, earth-abundant and highly efficient multi-functional electro-catalyst is very important for large scale hydrogen production by splitting water. In this work, the formed electro-catalyst shows porous structure which can provide a large surface area and active transfer of substance.

The over-potentials for both hydrogen evolution reaction and oxygen evolution reaction at the current density of 10 mA cm^{-2} in 1 M potassium hydroxide 213 mV and 324 mV respectively. For water splitting whole process, a small cell voltage of 1.63 V can achieve the required current density of 10 mA cm^{-2} in 1 M potassium hydroxide. This work will provide an observable guidance for the rational design and development of hetero-structure electro-catalysts for whole water splitting methods.



Finally, throughout the electrochemical process, particularly for OER at the anode, the reconstruction of surface structure and crystalline phase is unavoidable, which has lately emerged as a study focus on water electrolysis catalysts. Surprisingly, the self-reconstruction of the surface phase is often followed by a change in catalyst morphology into a layer-like structure, both of which increase the intrinsic activity of the original "pre-catalysts".

As a result, it is thought that the in situ produced (oxy) hydroxides derived from the "pre-catalyst" under OER work as the true active species to dominate water oxidation. For example, I developed NF-supported NiGe intermetallic compounds, which swiftly and thoroughly converted into OH/CO₃²⁻ intercalated γ -NiOOH under anodic activation, resulting in outstanding OER activity and durability in alkaline conditions. However, for most nanostructured catalysts, the reconstruction zone is often constrained to less than 10 nm at the surface. The dense near-surface rebuilt layer, which inhibits the exposure of active sites and limits further catalytic performance promotion, prevents interior oxidation.

To that end, deep, if not comprehensive, reconstruction of "pre-catalysts" has become a must-have concern for rational catalyst design. One solution to this challenge is to undertake an electrochemical activation process at a higher operating temperature. At a high activation temperature of 51.9°C, Mai et al. used the anode-CP technique to activate the "pre-catalyst" NiMoO₄ on NF, which was totally reassembled into the active NiOOH species. At the activation temperature of 25°C, low-degree reconstruction of the same "pre-catalyst" can be noticed. The rich grain boundaries and vacancies in the entirely recreated NiOOH nanoparticles reduced the reaction energy barrier, which explains the remarkable water splitting performance.

The third option is to reduce the bulk "pre-catalysts" to ultrafine nanounits. Another study by Mai et al. demonstrates that NiO nanosheets grown on NF (NiO/NF) were used as a "pre-catalyst." After lithiation breaks NiO into sub-10 nm ultra-small nanoparticles, CV at anode potential readily achieves deep reconstruction of NiO into active NiOOH with numerous defects.

Because of the hindered electrolyte penetration, only a 5 nm NiOOH layer formed on the NiO "pre-catalyst" after direct electro-oxidation. As a result, the deep reconstructed NiOOH demonstrated much higher OER activity, durability, and corrosion resistance in the alkaline electrolyte.

Conclusions

The tight bonding between Tb-CoB LDH and Tb-CoO ensures good electron transfer as Tb-CoB LDH-Tb-CoO exhibits outstanding and promising electro-catalytic property for both hydrogen evolution reaction and oxygen evolution reaction in an alkaline medium.

Finally, Tb-CoB LDH-Tb-CoO /terbium foam is designed through a stepwise process which is hydrothermal where commercial terbium foam is etched successfully by strong oxidizing agents to develop a self-supported electro-catalyst as the layered nano-structure of Tb-CoB LDH and Tb-CoO help to exhibit an outstanding catalytic activity.

Employing Tb-CoB LDH-Tb-CoO /terbium foam in a two-electrode alkaline medium for water splitting as an electrolyzer for a high intrinsic catalytic performance with excellent stability arises from a strong interaction among the components, faster charge transfer process, presence of abundant active sites and improved conductivity in the ternary electro-catalyst. The efficiency and duration of this designed cheap, lanthanide/transition metal based electrode are significant to meet the requirements of the industry as a self-supported water electro-catalyst. Hydrogen as a future source of energy and strategic emerging with environmental friend will be an essential part

of the international energy instead of fossil fuel and de-carbonization energy carrier as there is no carbon dioxide or monoxide emissions.

Getting hydrogen from water electro-catalyst benefits a large scale renewable energy consumption and energy structure transformation. To full the future increasing need of this source due to its high efficiency and long life water electrolyzers. Concepts of interfacial engineering and manufacturing methods are held to improve nano-technology industry which is trending and promising.

This research is based on interfacial engineering principles to summarize progress of self-supported electrodes with a concentration on enhancing the stability of the electrodes and activity of electro-catalysts to examine the effect of nano-structures on catalysts' performance especially at many interfaces. The future work will focus mainly on development self-supported electro-catalysts with higher activity and higher stability.

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