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$WO_3/BiVO_4$ photoanode coated with mesoporous Al_2O_3 layer for oxidative production of hydrogen peroxide from water with high selectivity†

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A WO $_3$ /BiVO $_4$ photoanode coated with various metal oxides demonstrated high selectivity (faradaic efficiency) for hydrogen peroxide (H $_2$ O $_2$) generation from water (H $_2$ O) under irradiation of simulated solar light in a highly concentrated hydrogen carbonate (KHCO $_3$) aqueous solution. A mesoporous and amorphous aluminium oxide (Al $_2$ O $_3$) layer significantly facilitated inhibition of the oxidative degradation of generated H $_2$ O $_2$ into oxygen (O $_2$) on the photoanode, resulting in unprecedented H $_2$ O $_2$ selectivity (ca. 80%) and the accumulation (>2500 μ M at 50C).

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Chemical conversions using light energy have been performed in various fields since the discovery of the Honda-Fujishima effect. 1-25 Significant efforts have recently been devoted to H₂ production by water splitting using inexhaustible light for clean energy conversion processes. 1-4,8-29 Photoelectrode systems are widely recognised as a promising technology for H2 production because they operate at an electrolysis voltage lower than the theoretical electrolysis voltage of water (<1.23 V).^{1,8-29} Visible light-responsive oxide photoanodes with a narrow bandgap energy, such as WO₃, BiVO₄ and Fe₂O₃, are desirable for the efficient utilisation of solar light and economical synthetic processes.8-29 Most importantly, numerous efforts have been focused on BiVO₄ photoanodes capable of utilising a wide range of light energy (\sim 520 nm) and achieving efficient O₂ generation by water splitting.9-21,28,29,32 A WO3/BiVO4 photoanode that combines BiVO₄ with a WO₃ underlayer for the efficient transfer of excited electrons on BiVO₄ to the F-doped SnO₂ conductive glass (FTO) substrate shows exceptional photoelectrochemical performance for water splitting into H_2 and O_2 . $^{10-13,17-20,28,29,32}$ However, most previous investigations, containing electrochemical reactions, focused solely on the recovery of H₂ energy generated on the cathode and little attention was paid to the recovery of the oxidation products simultaneously evolved during water splitting.24-31

 H_2O_2 is an especially versatile and clean oxidation product having the potential to generate instead of O_2 from H_2O (eqn (1)).

$$2H_2O \rightarrow H_2O_2 + 2H^+ + 2e^- (E(H_2O_2/H_2O) = +1.77 \text{ V vs. RHE})$$
 (1)

However, the accumulation of H_2O_2 , generated oxidatively is extremely difficult because degradation of H_2O_2 into O_2 also occurs easily and oxidatively in a conventional photoelectrochemical system *i.e.* the redox potential of H_2O_2 degradation is more negative than the redox potential of H_2O_2 production from H_2O (eqn (1) and (2)), resulting in low selectivity for oxidative H_2O_2 generation.

$$H_2O_2 \rightarrow O_2 + 2H^+ + 2e^- (E(O_2/H_2O_2) = +0.68 \text{ V } vs. \text{ RHE})$$
 (2)

Recently, we reported that a photoelectrochemical system combining the WO₃/BiVO₄ photoanode and aqueous electrolyte of KHCO₃ under CO₂ bubbling could achieve simultaneous generation and accumulation of H₂O₂ and H₂ from H₂O (eqn (3)).^{28,29} In this system, the aqueous electrolyte of KHCO₃ acts as an excellent oxidative catalyst for generating H₂O₂ from H₂O. Moreover, H₂O₂ could be produced at no external bias on both a WO₃/BiVO₄ photoanode (from H₂O) and an Au cathode (from O₂) *via* a two-photon process (eqn (4)).²⁹

$$2H_2O \rightarrow H_2O_2 + H_2$$
 (two-photon process) (3)

$$2H_2O + O_2 \rightarrow 2H_2O_2$$
 (two-photon process) (4)

Although the selectivity (faradaic efficiency: $\eta(H_2O_2)$) of reductive H_2O_2 production from O_2 on cathodes such as Au was very high, almost 100%, the maximum selectivity ($\eta(H_2O_2)$) for oxidative H_2O_2 production on WO₃/BiVO₄ photoanodes was still

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 $[\]dagger$ Electronic supplementary information (ESI) available: Experimental section, SEM images, XRD spectra, LHE spectra, applied voltage properties, $\it I-V$ characteristic of photoanodes, pore size distribution of the MeO_x particles, effect of Al₂O₃ amount on WO₃/BiVO₄ and dependence of applied voltage. See DOI: 10.1039/c7ra09693c

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low, only ca. 54%. The design of novel photoanodes capable of achieving efficient H₂O₂ generation and inhibiting oxidative degradation of generated H2O2 is absolutely imperative for building a clean and breakthrough technology, by accumulating H₂O₂ and H₂ with unprecedented H₂O₂ selectivity using only H₂O as the raw material.

Here, we focused on a surface modification of the metal oxide (MeO_r) layers on the WO₃/BiVO₄ photoanode surface to achieve excellent selectivity of generation and accumulation of H₂O₂ in the KHCO₃ aqueous solution under simulated solar light irradiation (Fig. 1). The MeO_x layers were prepared by spincoating of metal organic solutions and calcination. Introducing a porous Al₂O₃ layer was found to specifically permit oxidative H₂O₂ generation and accumulation with exceptional selectivity in an aqueous KHCO₃ electrolyte because of the blocking effect of oxidative degradation of the generated H2O2 into O2 on the photoanode.

Details regarding experimental procedures for preparation and photoelectrochemical reactions of photoanodes are provided in the ESI†.

The effects of MeOx layers, modified on the WO3/BiVO4 photoanode, for oxidative H2O2 generation properties were investigated at an applied electric charge of 0.9C (900 s at steady photocurrent of 1 mA) in a 0.5 M KHCO₃ aqueous electrolyte. As shown in Fig. 2, all MeO_x-coated photoanodes, except CoO_x, enhanced the oxidative H2O2 generation compared to a bare WO₃/BiVO₄ photoanode, and the enhanced effect, ranked by the modified metal oxide, was $Al_2O_3 > ZrO_2 > TiO_2 > SiO_2 >> CoO_r$. Little H_2O_2 was observed on the CoO_x coated photoanode, because CoO_x probably decomposed the generated H₂O₂ quickly, or O_2 may be evolved on CoO_x directly. It should be noted that the Al₂O₃ modification on the WO₃/BiVO₄ photoanode achieved roughly twice the oxidative H₂O₂ generation compared to the bare WO₃/BiVO₄ photoanode. The Al₂O₃ uniformly, smoothly and flatly covered the entire area of the WO₃/BiVO₄ photoanode as shown in the SEM images (Fig. 3), whereas other MeO_x were granularly and uniformly supported on that and possessed any crack holes (Fig. S1; ESI†). It was also confirmed, from XRD measurement (Fig. S2; ESI†), that no

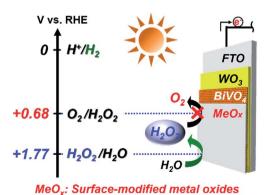


Fig. 1 Pattern and energy diagrams for photoelectrochemical H₂O₂ generation from H₂O on WO₃/BiVO₄/MeO_x photoanodes under solar light irradiation.

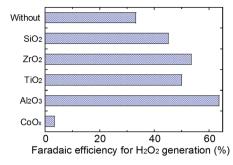


Fig. 2 Oxidative H₂O₂ generation on photoanodes (WO₃/BiVO₄/ MeO_x) modified various metal oxides on a WO₃/BiVO₄ at an electric charge of 0.9C (900 s at steady photocurrent of 1 mA) in a 0.5 M KHCO₃ aqueous electrolyte (35 mL) in an ice bath (below 5 °C) under simulated solar light.

diffraction peaks derived from MeO_r were observed in all WO₃/ BiVO₄/MeO_r photoanodes, suggesting that all tried MeO_r modified on WO₃/BiVO₄ photoanode possess amorphous-like structure. As shown in Fig. S3; ESI,† little change of the light harvesting efficiency (LHE) was also confirmed in tried all photoanodes, suggesting that these MeOx introduced on the WO₃/BiVO₄ have little effect to light absorption efficiency on WO₃/BiVO₄ photoanode. The time courses of voltages applied between photoanode and a counter electrode of Pt mesh at steady photocurrent of 1 mA (Fig. 2) in oxidative H2O2 generation reaction were also confirmed (Fig. S4; ESI†). The voltages for applying steady photocurrent of 1 mA slightly increased by introducing MeO_x on the WO₃/BiVO₄ photoanode. In particular, WO₃/BiVO₄/Al₂O₃ photoanode, coated uniformly, smoothly and flatly at Al₂O₃ compared to other MeO_x, required highest applied voltage. In order to confirm the effect introducing the Al₂O₃ on the photoanode in more detail, the photocurrent property of the WO₃/BiVO₄/Al₂O₃ photoanode was investigated in a 0.5 M KHCO₃ aqueous solution (Fig. S5; ESI†). The bare WO₃/BiVO₄ photoanode exhibited excellent photocurrent property in all applied voltage ranges as with our past reported example, 11,12,28,29 and the photocurrent property slightly decreased by introducing the Al₂O₃ layer. However, it should be noted that the decreasing degree of the photocurrent property was slight, only ca. 9% and 5% at +1.23 V and +1.77 V vs. RHE, respectively, although the Al2O3, having an insulation property, covered the entire area of the WO₃/BiVO₄ photoanode. A similar

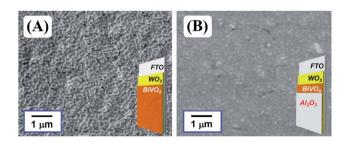


Fig. 3 SEM images of (A) WO₃/BiVO₄ and (B) WO₃/BiVO₄/Al₂O₃ photoanodes

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phenomenon has also been observed in O2 and H2 generation through water splitting on a photoanode coated with amorphous-like Ta2O5.32 In addition, it was confirmed, from the N₂ absorption and desorption measurement of MeO_x particles (Fig. S6; ESI \dagger), that almost all MeO_x possess mesoporous structure at a pore size of ca. 4-20 nm. In particular, a pore size of the Al₂O₃ was ca. 4.7 nm. The thicknesses of Al₂O₃ calculated from the coating amount on the WO₃/BiVO₄ photoanode by XRF measurement were ca. 100 nm $(0.055 \text{ mg cm}^{-2})$. In order to also investigate the effects of dense Al2O3 on WO3/BiVO4 on the oxidative H₂O₂ generation, increasing Al₂O₃ amount on WO₃/ BiVO₄ photoanode was performed by decreasing the spin coating number (500 rpm) of precursor solution of EMOD solved in butyl acetate containing ethylcellulose when introducing Al₂O₃ layers. The thickness of Al₂O₃ introduced at 500 rpm calculated from the XRF measurement was ca. 127 nm (0.070 mg cm⁻²), suggested that the thickness increases with decreasing the spin coating number. As shown in Fig. S7; ESI,† little change of the H₂O₂ generation amounts was observed on these WO₃/BiVO₄/Al₂O₃ photoanodes prepared at 500 and 1000 rpm, indicating that increasing Al₂O₃ on WO₃/BiVO₄ photoanode has little effect on the oxidative H₂O₂ generation. In subsequent experiments, WO₃/BiVO₄/Al₂O₃ prepared at 1000 rpm was utilized as the photoanode. These results indicate that the specific effect enhancing oxidative H₂O₂ generation property was achieved on the WO₃/BiVO₄ though the mesoporous and amorphous Al₂O₃ layer covered uniformly, smoothly and flatly the entire area.

To track the specific performance enhancing effect of generating H₂O₂ by introducing the Al₂O₃ layer, the concentration dependency of KHCO₃ aqueous electrolytes on the oxidative H2O2 generation property was investigated at an applied electric charge of 0.9C (Fig. 4(A)). We have already reported that the oxidative H₂O₂ generation property on the WO₃/ BiVO₄ photoanode was improved with increasing concentration of KHCO3, which acts as an effective catalyst for H2O2

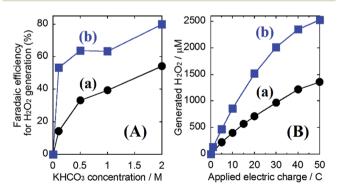


Fig. 4 (A) Oxidative H₂O₂ generation in KHCO₃ aqueous electrolytes (35 mL) of different concentrations at applied electric charges of 0.9C (900 s at steady photocurrent of 1 mA) under simulated solar light and (B) accumulation of oxidative H₂O₂ generation in a 2.0 M KHCO₃ aqueous solution (35 mL) under visible light irradiation ($\lambda > 420$ nm) using an intense Xe lamp at an applied voltage of 1.5 V in an ice bath (below 5 °C) on a (a) bare WO₃/BiVO₄ and (b) WO₃/BiVO₄/Al₂O₃ photoanodes

generation via the two-electron oxidation of H2O.28 Even in the case of using the WO₃/BiVO₄/Al₂O₃ photoanode, the selectivity $(\eta(H_2O_2))$ for H_2O_2 generation was significantly enhanced with increasing concentration of KHCO₃, and the $\eta(H_2O_2)$ in the 2.0 M KHCO₃ agueous solution reached ca. 80% at 0.9C, whereas that using the bare WO₃/BiVO₄ photoanode was ca. 54%. It should be noted that the selectivity $(\eta(H_2O_2) = ca. 53\%)$ on the WO₃/BiVO₄/Al₂O₃ photoanode in lowly concentrated KHCO₃ (0.1 M) was comparable to that (ca. 54%) on the bare WO₃/BiVO₄ photoanode in highly concentrated KHCO₃ (2.0 M). This suggests that the Al₂O₃ could effectively be contributing to oxidative H2O2 generation from H2O even in the lowly concentrated KHCO₃. Moreover, as shown in Fig. 4(B), the excellent H₂O₂ generation property on the WO₃/BiVO₄/Al₂O₃ photoanode compared to the WO₃/BiVO₄ photoanode was significantly maintained even at high electric charge up to 50C. As a result, the accumulation amount, using the WO₃/BiVO₄/Al₂O₃ photoanode, reached >2500 μM at 50C, while that using the bare WO₃/ BiVO₄ photoanode was >1300 μ M at 50C. The dependency of the applied voltage on the oxidative H2O2 generation was investigated to confirm the effect of the Al₂O₃ coating in detail (Fig. S8; ESI†). A small change in H₂O₂ generation performance was observed in all ranges of applied voltages (0.8-1.8 V), suggesting that the enhanced effect of introducing an Al2O3 layer is independent of the voltages applied between a photoanode as the working electrode and a Pt mesh as counter electrode using the aqueous electrolyte of the KHCO₃.

Although little development with regards to highly selective H₂O₂ generation via two-photon oxidation of H₂O and accumulation using photoanodes has been reported, our method of Al₂O₃ coating on the WO₃/BiVO₄ photoanode produced tremendous improvement in selective H2O2 generation and accumulation from H₂O in a KHCO₃ aqueous electrolyte. It is speculated that the specific enhancement of selectivity for H2O2 generation on the WO₃/BiVO₄/Al₂O₃ photoanode may be caused by a blocking effect, on the mesoporous Al₂O₃ layer, that inhibits oxidative H₂O₂ degradation into O₂ on the BiVO₄. To investigate the blocking effect on the Al₂O₃ layer, a degradation property test of H₂O₂ was performed in a 2.0 M KHCO₃ aqueous solution containing H_2O_2 (550 μ M) in the presence of the bare WO₃/BiVO₄ or WO₃/BiVO₄/Al₂O₃ photoanodes in presence or absence of simulated solar light irradiation in an ice bath (below 5 °C). In both cases, as shown in Fig. 5, almost all the initial amount of H₂O₂ was maintained in the dark condition, however, the H2O2 amount drastically decreased with irradiation by simulated solar light, suggesting that the H₂O₂ was decomposed by photocarriers (excited electrons and holes) produced on the BiVO₄. It should be noted that the H₂O₂ degradation in the presence of the WO₃/BiVO₄/Al₂O₃ photoanode was dramatically inhibited compared to the degradation in the presence of a bare WO₃/BiVO₄ photoanode. The oxidative H₂O₂ generation test was also confirmed in a 2.0 M KHCO₃ aqueous electrolyte, initially containing H_2O_2 (210 μ M) on the bare WO₃/BiVO₄ and WO₃/BiVO₄/Al₂O₃ photoanodes, to track the generated H₂O₂ degradation behaviour in more detail (Fig. 6). The generated rates of H₂O₂ were reduced by the initial addition of H₂O₂ in both cases of presence or absence of Al₂O₃.

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Solar light OFF Solar light ON (a)

Gradation properties of H_2O_2 (550 μ M) initiall CO_3 aqueous solution in an ice bath (below 5 °C) and simulated solar light irradiation in the prese

Fig. 5 Degradation properties of H_2O_2 (550 μM) initially added in a 2.0 M KHCO $_3$ aqueous solution in an ice bath (below 5 °C) under CO_2 bubbling and simulated solar light irradiation in the presence of a (a) $WO_3/BiVO_4$ and (b) $WO_3/BiVO_4/Al_2O_3$ photoanodes at no applied voltage.

However, the decreasing rate of H₂O₂ generation was significantly inhibited, from ca. 61% to ca. 39%, by introducing the Al₂O₃ layer on the WO₃/BiVO₄ photoanode. These results suggest that introducing the Al₂O₃ layer significantly contributed to the highly selective H₂O₂ generation and accumulation from H₂O, with a high photocurrent property, by a blocking effect that inhibited the oxidative degradation of generated H₂O₂. The mechanism of blocking effect is proposed that the H₂O₂ generated on the BiVO₄ in the WO₃/BiVO₄/Al₂O₃ photoanode diffuses in electrolyte of KHCO3 aqueous solution through mesoporous of the Al₂O₃, and contact of the H₂O₂ diffused in electrolyte with the BiVO4 covered uniformly and smoothly Al₂O₃ may be significantly inhibited compared with that with bare BiVO₄, resulting in the formation of effective inhibition of oxidative H₂O₂ degradation. Furthermore, there may be other possible mechanisms such as a blocking effect of a direct O₂ evolution site via a 4-photon process covering by Al₂O₃, or an enrichment effect resulting from the increasing KHCO₃ concentration around the photoanode based on the acid-base adsorption between HCO3- (a weak base) and the weakly acidic sites on the Al₂O₃ surface, related to the good

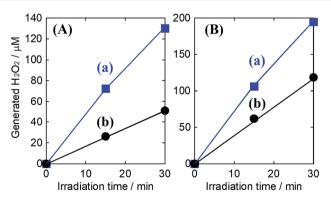


Fig. 6 Comparison of oxidative H_2O_2 generation in a 2.0 M KHCO3 aqueous electrolyte (a) in the absence of or (b) containing initially-added H_2O_2 (210 $\mu\text{M})$ in an ice bath (below 5 °C) on a (A) WO3/BiVO4 and (B) WO3/BiVO4/Al2O3 photoanodes under simulated solar light irradiation at steady photocurrent of 1 mA.

 $\eta(\mathrm{H_2O_2})$ in lower KHCO₃ concentration, as shown in Fig. 4(A). The tracking and contribution of these other mechanisms, on the $\mathrm{Al_2O_3}$ layer, is currently under investigation.

Conclusions

In summary, various metal oxides were coated onto a WO₃/ BiVO₄ photoanode to enhance the selectivity (faradaic efficiency) of oxidative H₂O₂ generation, in an aqueous electrolyte of KHCO3, from water under solar light irradiation. Among the various metal oxides, the Al₂O₃ coating, which produced a mesoporous and amorphous structure on the WO₃/BiVO₄ photoanode, achieved excellent oxidative H2O2 generation at a selectivity of ca. 80% and an accumulation of >2500 μ M (50C). Interestingly, the Al₂O₃-coated WO₃/BiVO₄ photoanode dramatically inhibited oxidative degradation of H₂O₂ generated on the WO₃/BiVO₄ photoanode after introducing the Al₂O₃ layer. This study contributes to developing a promising design for a clean H₂O₂ production system that uses only water as the raw material under solar light irradiation. More effective dreamy H2O2 generation, at an excellent selectivity close to 100%, can be expected by modifying the surface-treatment technology, and it is currently under investigation.

Conflicts of interest

There are no conflicts to declare.

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