Experimental Procedures
**Chemicals**

Ni(acac)$_2$ (95%), 1-octadecene (90%), Nafion perfluorinated resin solution (5 wt%), VO(acac)$_2$ (98%), KI (99+%), and p-benzoquinone (>98%) were purchased from Sigma-Aldrich Co., LLC. Oleylamine (C18 content 80–90%) was purchased from Acros Organics. Fe(CO)$_5$ (>95%) and tri-n-octylphosphine (TOP, >95%) were purchased from Kanto Chemical Co., Inc. Bi(NO$_3$)$_3$·5H$_2$O (99.9%) was purchased from Wako Pure Chemical Industries, Ltd. Di-n-octyl ether (>95%) was purchased from Tokyo Chemical Industry Co., Ltd. All reagents were used without further purification.

**Characterization**

Transmission electron microscope (TEM) observations were performed on a JEM1011 (JEOL) operated at 100 kV. High-resolution TEM (HRTEM) and scanning TEM-energy dispersive X-ray spectroscopy (STEM-EDS) observations were performed on a JEM-ARM200F operated at 200 kV. Scanning electron microscope (SEM) observations were performed on a S-4800 (Hitachi). X-ray diffraction (XRD) patterns were measured on PANalytical X’Pert Pro MPD with CuKα radiation ($\lambda = 1.542$ Å) operated at 45 kV and 40 mA. X-ray photoelectron spectroscopy (XPS) measurements were conducted in an ultrahigh vacuum combined system equipped with a hemispherical electron analyzer and a Mg Kα X-ray source (1253.6 eV). The detail has been described in a previous paper[1]. Transmittance measurements were acquired on a U-3310 spectrophotometer (Hitachi). X-ray fluorescence (XRF) elemental analysis was performed on an Element Analyzer JSX-3202C (JEOL). Fourier Transform infrared (FT-IR) analyses were performed on a IR Prestige 21 (Shimadzu).

**Synthesis of a-NiP$_x$ NPs**

Ni(acac)$_2$ (1 mmol), 1-octadecene (4.5 mL), and oleylamine (6.4 mL) were mixed and heated to 80 °C under a N$_2$ atmosphere to form a blue-green transparent solution. TOP (2 mL) was injected and the mixture was heated to 230 °C at 10 °C min$^{-1}$, and maintained at that temperature.
for 30 min. The reaction solution was then cooled to room temperature and purified with ethanol. The precipitated solid NPs were redispersed in hexane and stored for further use.

**Synthesis of NiPₓ@FePₓOᵧ NPs**

A 34-mg portion of a-NiPₓ NPs was dispersed in a mixture of 1-octadecene (9 mL) and oleylamine (1.9 mL). After repeated degassing-refilling with N₂, TOP (2 mL) solution containing Fe(CO)₅ (0.4 mmol) was injected into the mixture at room temperature. The mixed solution was heated to 270 °C for 60 min. After the reaction, the solution was cooled to room temperature and purified with ethanol. The precipitated solid NPs were redispersed in hexane and stored for further use.

**Synthesis of NiₓP NPs**

a-NiPₓ NPs (85 mg) was dissolved in a mixture of di-n-octyl ether (9 mL) and oleylamine (1.9 mL). The mixture was heated to 270 °C at 10 °C min⁻¹ under a N₂ atmosphere. The solution was reacted at 270 °C for 60 min, cooled to room temperature, and purified with ethanol. The precipitated solid NPs were redispersed in hexane and stored for further use.

**Synthesis of FeOₓ NPs**

The 5-nm FeOₓ NPs were synthesized following a literature procedure [2]. Briefly, a mixture of Fe(CO)₅ and oleylamine (1:1 mol/mol) was heated to 80 °C at 2 °C min⁻¹ and held at that temperature for 30 min under a N₂ atmosphere. A deep red solution of Fe(CO)₅-oleylamine complex was injected into 1-octadecene at 180 °C in a N₂ atmosphere. The solution was reacted at 180 °C for 30 min, cooled to room temperature, and then purified with ethanol. The precipitated solid NPs were redispersed in hexane and stored for further use.

**Preparation of NP-loaded carbon powder catalysts**

The electrocatalyst powder was prepared by mixing NPs and conductive carbon powder (XC-72, Cabot). Typically, XC-72 (8 mg) was dispersed in hexane (8 mL) by sonication, followed by the dropwise addition of hexane (2 mL) containing NPs (2 mg). Further sonication was
applied for 30 min. The catalyst powder was collected by centrifugation, washed with acetone twice, and dried under vacuum.

**Preparation of carbon powder catalyst working electrodes**

Catalyst powder (1 mg) was mixed with water (396 µL), 2-propanol (94 µL), and Nafion resin solution (10 µL), then sonicated for 30 min to form a homogenous catalyst slurry. The catalyst slurry was deposited on a glassy carbon rotating disk electrode 5 mm in diameter and dried by rotating at 700 rpm under ambient conditions.

**Preparation of NP-loaded carbon paper electrodes**

A 1 × 3 cm² piece of carbon paper (Toray, TGP-H-120) was washed with ethanol and acetone, and dried under vacuum. A hexane solution of NiPₓ@FePᵧOᵦ NPs was dropped on the carbon paper (0.01, 0.075 or 0.5 mg cm⁻²), washed with ethanol, and dried under vacuum.

**Immobilization of NPs on FTO-coated glass electrodes**

A 1.5 × 5 cm² piece of FTO-coated glass (AGC Fabritech) was washed with ethanol and acetone. The hexane solution of NiPₓ@FePᵧOᵦ NPs (3 mg mL⁻¹, 50 µL) was dropped onto the FTO coated glass and spun at 1000 rpm for 10 s (the coated area was 2.25 cm²).

**Acid etching of NiPₓ@FePᵧOᵦ NPs**

The NiPₓ@FePᵧOᵦ NPs-loaded carbon powder was mixed with 0.5 M H₂SO₄ aqueous solution (2 mL) and sonicated for 1 h. Then the powder was collected by centrifugation and washed with water and acetone. Finally, the powder was dried under vacuum for TEM and XRF measurements.

**Preparation of BiVO₄ electrodes**

We prepared porous BiVO₄ electrodes according to the method reported by Choi et al. [3]. Briefly, a BiOI film was deposited on an FTO coated glass substrate by immersing the substrate in a water/ethanol solution of Bi(NO₃)₂, KI, and p-benzoquinone, followed by the application of −0.1 V vs Ag/AgCl (3 M KCl) for 2 min (the deposited area was 1~1.5 cm²). A DMSO solution of VO(acac)₂ was placed on the BiOI film, followed by annealing at 450 °C for 2 h in
air. The annealed electrode was immersed in 1 M NaOH for 30 min to remove V₂O₅ by-products, washed with deionized water, and dried under ambient conditions.

**NP deposition on BiVO₄ electrodes**

Before loading the NPs on the BiVO₄ photoelectrodes, photoelectrochemical (PEC) measurements of all BiVO₄ photoelectrodes were performed in 0.125 M K₂B₄O₇ (pH 9.4) with a 300 W Xe lamp with a >385 nm short-wavelength cut-off filter (see below), and photoelectrodes showing the similar photocurrents were selected for NP loading for comparison with the photocurrents precisely. The FTO surface of the BiVO₄ electrode was masked with scotch tape. A hexane solution of NPs (0.05, 0.25, 1 or 5 mg mL⁻¹, 25 µL) was dropped on the BiVO₄ film, and spun at 1000 rpm for 10 s. The NP-deposited electrodes were washed with ethanol and dried under ambient conditions.

**Electrochemical measurements**

The electrochemical measurements were performed in a three-electrode electrochemical cell with a ALS620C electrochemical analyzer (BAS) at room temperature (~25 °C). Hg/HgO (1 M NaOH) or Ag/AgCl (3 M NaCl) was used as a reference electrode in 0.1 M KOH or 0.125 M K₂B₄O₇ electrolyte, respectively. The reference electrodes were calibrated with an unused saturated calomel electrode in saturated NaCl aqueous solution. A Pt coil was used as a counter electrode. Prior to each measurement, the electrolyte was deaerated by bubbling with Ar for 20 min to remove oxygen. Cyclic voltammograms and chronoamperometry were conducted under a continuous Ar flow. In the case of a rotating disk electrode, the working electrode was rotated at 1600 rpm during measurements with a RRDE-3A (BAS). In the case of carbon paper and FTO coated glass electrodes, the working electrode was immersed and the electrolyte solution was stirred during the measurements. All displayed voltammograms except for Figure S9 and S11b were iR-collected to account for any uncompensated resistance. The uncompensated resistance was measured with a function of the analyzer based on a previously reported
technique [4]. The conversion between potentials vs. reference electrode ($E_{\text{ref}}$) and vs. reversible hydrogen electrode (RHE) ($E_{\text{RHE}}$) was performed with the equation below:

$$E_{\text{RHE}} = E \text{ (vs. } E_{\text{ref}}\text{)} + E_{\text{ref}} + 0.059 \times \text{pH} - iR$$

**PEC measurements**

The PEC performance of the photoanodes was evaluated in a three-electrode configuration with a ALS620C electrochemical analyzer (BAS) and a light source (300 W Xe lamp, MAX-303, Asahi Spectra). Illumination was obtained by passing light from the source through a >385 nm short wavelength cut-off filter, and irradiated through the FTO side (back-side illumination). The power density of the incident light was ~190 mW cm$^{-2}$ at the BiVO$_4$/FTO electrode measured by a thermal sensor (PS10, Coherent). The simulated sunlight was obtained using 300 W Xe lamp equipped with AM filter. Power density was adjusted to 100 mW cm$^{-2}$ using standard Si solar cell (BS-500BK, BUNKOUKEIKI). Photocurrent measurements were performed in 0.125 M K$_2$B$_4$O$_7$ buffer solution (pH 9.4). Prior to each measurement, the electrolyte was deaerated by bubbling Ar for 20 min. Photocurrents were obtained with illumination and sweeping the potential in the positive direction at a scan rate of 10 mV s$^{-1}$ or applying a constant bias. All measurements were performed with a Ag/AgCl (3 M KCl) reference electrode, and all results in this work are reported against RHE. The Ag/AgCl reference electrode was calibrated with an unused saturated calomel electrode in saturated NaCl solution. The conversion between potentials vs. Ag/AgCl and vs. RHE was performed using the equation below:

$$E_{\text{RHE}} = E \text{ (vs. } \text{Ag/AgCl}\text{)} + E_{\text{Ag/AgCl}} + 0.059 \times \text{pH}$$
The surface charge transfer efficiency ($\eta_{\text{surface}}$) using the photocurrent of water ($J_{\text{water}}$) and sulfite ($J_{\text{sulfite}}$) oxidation according to the equation below.

$$\eta_{\text{surface}} \% = \frac{J_{\text{water}}}{J_{\text{sulfite}}} \times 100\%$$
Fig. S1 Additional HRTEM images of NiP₃@FeP₂O₅ NPs.

Fig. S2 (a) TEM images and (b) XRD patterns of NiP₃@FeP₂O₅ NPs at various reaction time.
Fig. S3 Temporal change of Fe/Ni molar ratios of NiP₃@FeP₃O₂ NPs in the synthesis measured by XRF.

Fig. S4 (a) TEM image and (b) XRD pattern of NPs synthesized without Fe(CO)₅.
Fig. S5 (a) Change in (111) diffraction peak positions of Ni$_{2-x}$Fe$_x$P phase depending on $x$ using the data in [5]. (b) (111) diffraction patterns of Ni$_2$P NPs and NiP$_x$@FeP$_y$O$_z$ NPs in this work.
**Fig. S6** TEM images of (a) NiP$_x$@FeP$_y$O$_z$ NPs and (b) shell-etched NiP$_x$ cores supported on carbon powder. (c) XRF spectra of NiP$_x$@FeP$_y$O$_z$ NPs (blue) before and (red) after etching. Inset shows the corresponding Ni:Fe molar ratios.
Fig. S7 (a, b) TEM images and (c, d) XRD patterns of (a, c) Ni$_2$P NPs and (b, d) FeO$_x$ NPs.
**Fig. S8** TEM images of (a) Ni$_2$P NPs, (b) FeO$_x$ NPs, and (c) the mixture of Ni$_2$P+FeO$_x$ NPs supported on carbon powder.
**Fig. S9** Cyclic voltammograms of NiP$_x$@FeP$_y$O$_z$, FeO$_x$, Ni$_2$P, and FeO$_x$+Ni$_2$P NPs supported on carbon powder in 0.1 M KOH at 10 mV s$^{-1}$ without iR compensation.

**Fig. S10** Cyclic voltammograms of the NiP$_x$@FeP$_y$O$_z$ NPs at Ni/Fe molar ratio of 78/22 (black), 85/15 (red), and 72/28 (blue). The NiP$_x$@FeP$_y$O$_z$ NPs at Ni/Fe molar ratio of 85/15 78/22, and 72/28 were synthesized at 270 °C for 20, 60, and 120 min, respectively.
**Fig. S11** Cyclic voltammograms of bare and NiP₃@FeP₅O₂ NPs loaded carbon papers (a) with and (b) without $iR$ compensation. (c) Current density-time curves of NiP₃@FeP₅O₂ NPs loaded carbon paper at 1.9 V vs RHE without $iR$ compensation. Electrolyte: 0.1 m KOH, RE: Hg/HgO, CE: Pt coil.
**Fig. S12** (a) Cyclic voltammograms of the NiPₓ@FePᵧOلزم NPs supported on carbon paper at 1ˢᵗ and 100ˢᵗ cycle in 0.1 M KOH. (b) XRF spectra of the NiPₓ@FePᵧOلزم NPs supported on carbon paper (blue) before and (red) after 100 cycles CV in 0.1 M KOH. A table shows the molar ratios of Ni:Fe:P and the molar ratios of Ni:Fe in parentheses.

|         | preOER | postOER |
|---------|--------|---------|
| Ni      | 39 (81)| 74 (82) |
| Fe      | 12 (19)| 17 (18) |
| P       | 49     | 9       |
Fig. S13 XPS spectra of the NiP₃@FeP₂O₆ NPs supported on carbon paper before and after 100 cycles OER in 0.1 M KOH. (a) Ni 2p, (b) Fe 2p, (c) P 2p, (d) O 1s. Each binding energy was calibrated by C 1s peak (284.6 eV). The Ar bombardment was carried out at 500 eV of the beam energy for 2 min.
Effect of Ar bombardments

(a) Ni 2p: Before OER; after Ar bombardment, strong peaks of Ni$^0$, which corresponded to Ni in the NiP$_x$ core appeared. After OER; the Ar bombarded sample also showed Ni$^0$ peaks, which were attributed to reduced Ni cations in NiOOH formed during the Ar bombardment.

(b) P 2p: Before OER; after Ar bombardment, P$^0$ peak at 130 eV became strong owing to the exposure of P$^0$ in NiP$_x$ cores. After OER; no peaks appeared even after Ar bombardment, indicating P was not only absent from the surface but was also eliminated from the bulk during OER.

(c) Fe 2p: Before OER; after Ar bombardment, a weak Fe$^0$ peak emerged at 707.5 eV, which was attributed to Fe(0) in NiP$_x$ cores. After OER; no peaks of Fe$^0$ appeared because most of the NiP$_x$ cores were oxidized during OER.

(d) O 1s: Before OER; after Ar bombardment, O1s peak slightly shifted. One explanation for this shift is that the outer and inner parts of the shell were mainly oxide (531 eV) and phosphate (532 eV), respectively. After OER, after Ar bombardment, a shoulder peak at a lower binding energy (530 eV) assigned to metal oxide appeared, indicating that the inner part of the sample after OER contained metal oxide and (Ni, Fe)O$_x$H.
**Fig. S14** SEM images of the NiP$_x$@FeP$_y$O$_z$ NPs supported on carbon paper (a) before and (b) after 100 cycles CV in 0.1 M KOH.

**Fig. S15** (a) Cyclic voltammograms of the NiP$_x$@FeP$_y$O$_z$ NPs loaded on FTO coated glass at 1$^{\text{st}}$ and 30$^{\text{th}}$ cycle in 0.1 M KOH.
Fig. S16 (a) Cross sectional SEM image of the BiVO₄ photoelectrode. TEM images of (b) FeOₓ NPs, (c) Ni₂P NPs, (d) the mixture of Ni₂P+FeOₓ NPs, and (e) NiPₓ@FePᵧOz NPs loaded on BiVO₄ photoelectrodes.
Fig. S17 Current density durability of bare and NPs loaded BiVO₄ in CA measurements @1.23 V vs RHE.
Fig. S18 Photocurrent density curves of (a) bare BiVO$_4$ and (b) FeO$_x$, (c) Ni$_2$P, (d) Ni$_2$P+FeO$_x$ and (e) NiP$_x$@FeP$_2$O$_5$ NPs loaded BiVO$_4$ before and after CA measurements.
**Fig. S19** (a) Cyclic voltammograms and (b) XRF spectra of the NiP$_x$@FeP$_y$O$_z$ NPs supported on carbon papers at 0, 100, and 1000 cycles in 0.125 M $\text{K}_2\text{B}_4\text{O}_7$ at 100 mV s$^{-1}$. A table shows the molar ratios of Ni:Fe:P and the molar ratios of Ni:Fe in parentheses. Decreased P molar ratio after CV suggests that the NiP$_x$@FeP$_y$O$_z$ NPs transformed into the (Ni, Fe)O$_x$H$_y$ during OER even in 0.125 M $\text{K}_2\text{B}_4\text{O}_7$. 

| mol%  | 0 cyc | 100 cyc | 1000 cyc |
|-------|-------|---------|----------|
| Ni    | 38 (81) | 48 (79) | 73 (79)  |
| Fe    | 12 (19) | 16 (21) | 19 (21)  |
| P     | 50    | 36      | 8        |
**Fig. S20** (a) LSV of bare and various amounts of NiP₆@FeP₃O₂ NPs loaded BiVO₄ under simulated sunlight (100 mW cm⁻²). (b) Charge transfer efficiency of samples A and E. Current-time curves of BiVO₄ electrodes after (c) 100 s and (d) 3 h (10800 s) at 1.23 V vs RHE. (A) Bare and (B) 0.05, (C) 0.25, (D) 1, and (E) 5 mg mL⁻¹ NPs ink spin-casted BiVO₄. Solid lines: in 0.125 M K₂B₄O₇, Dashed lines: in 0.5 M potassium phosphate buffer with 1 M Na₂SO₃, RE: Ag/AgCl, CE: Pt coil.
**Table S1** Comparison of OER activities of various transition metal phosphide based OER electrocatalysts

| Catalyst | Morphology | Electrolyte | Overpotential @10 mA cm$^{-2}$/ V | loading amount / mg cm$^{-2}$ | Ref. |
|----------|------------|-------------|----------------------------------|-------------------------------|------|
| NiP$_x$@FeP$_y$O$_z$ on carbon powder | ~20 nm NP | 0.1 M KOH | 0.28 | 0.02 | This work |
| NiP$_x$@FeP$_y$O$_z$ on carbon paper | urchin shape 80 x 5 nm | 0.1 M KOH | 0.37 | 0.10 | [6] |
| CoFeP | 20 nm rods | 0.1 M KOH | 0.36 | 0.05 | [7] |
| CoMnP | ~5 nm NP | 1 M KOH | 0.33 | 0.284 | [8] |
| CoP | 1-5 μm needle film | 1 M KOH | 0.27 | 4.95 | [9] |
| NiFeP | a few μm thick film | 1 M KOH | 0.28 | a few mm thickness | [10] |
| Ni phosphides | >300 nm nanoporous solid | 1 M KOH | 0.32 | 0.15 | [11] |
| Ni$_5$P$_4$ | 40 μm thick film | 1 M KOH | 0.29 | 11.71 | [12] |
| Ni$_3$P | 50 nm solid | 1 M KOH | 0.29 | 0.14 | [13] |
| Fe doped Ni$_3$P | 1 μm nanosheet | 1 M KOH | <0.2 | 1.4 | [14] |
| NiFe-OH/NiFeP | several μm nanosheet | 1 M KOH | 0.199 | 1.8 | [15] |
| FeO$_x$@NiP$_y$ | ~10 nm NP | 1 M KOH | 0.26 | 0.2 | [16] |
| GDs/Co$_3$Ni$_2$:P | ~200 nm porous tubular | 1 M KOH | 0.287 | no data | [17] |
| Mg/Ni$_3$P | thin film | 1 M KOH | 0.28 | 1.33 | [18] |
| FeP$_z$FeP$_y$O$_z$ | 100 x 800 nm hollow | 1 M KOH | 0.28 | 0.3 | [19] |
| Co$_{0.5}$Fe$_{0.5}$P$_{1.1}$ | 200-500 nm sphere | 1 M KOH | 0.21 | 0.2 | [20] |
| NiFe(3:1)-P | flower-like 1-1.5 μm | 1 M KOH | 0.233 | 0.1 | [21] |
### Table S2 Comparison of performance of cocatalyst-modified nondoped-BiVO₄ photoelectrode under simulated sunlight.

| Cocatalyst                  | Deposition method          | Photocurrent @1.23V/mA cm² | Photocurrent retension | Ref.  |
|-----------------------------|-----------------------------|-----------------------------|------------------------|-------|
| CoPi                        | PED⁺                        | 6.1                         | 90% at 0.6 V RHE in 6h | [22]  |
| Co₃O₄ Cubane                | dropcasting                 | 5                           | ~50% at 0.7 V RHE in 4h | [23]  |
| β-FeOOH                     | impregnation                | 4.3                         | 100% at 1 V RHE in 2h  | [24]  |
| FeOOH                       | PED⁺                        | 4.2                         | 100% at 0.6 V vs CE in 48h | [25] |
| CoOOH                       | nanosheet dropcasting       | 4                           | 97% at 1 V RHE in 4h   | [26]  |
| NiB                         | NPs dropcasting             | 3.47                        | ~50% at 1.23 V RHE in 1.1h | [27] |
| In₃O₅/CoOOH                 | CBD⁺+PEC deposition         | 3.4                         | ~85% at 1.23 V RHE in 1h | [28] |
| ZnFe₂O₄/Co²⁺               | PED⁺+immersion              | 2.84                        | ~75% at 1.23 V RHE in 0.83h | [29] |
| ZnFe₂O₄                    | PED⁺                        | 2.76                        | ~60% at 1.23 V RHE in 0.83h | [29] |
| Co²⁺                       | immersion                   | 1.53                        | ~10% at 1.23 V RHE in 0.83h | [29] |
| Co₃O₄                       | NPs dropcasting+annealing   | 2.71                        | ~80% at 0.6 V RHE in 0.1h | [30] |
| Ni:Co₃O₄                   | electrospray pyrolysis      | 2.62                        | ~40% at 0.77 V RHE in 2h | [31] |
| NiP/Fe₃P₂O₅                 | NPs dropcasting             | 2.32                        | 17% at 1.23 V RHE in 3h | this work |
| NiP/Fe₃P₂O₅                 | NPs dropcasting             | 2.28                        | 46% at 1.23 V RHE in 3h | this work |
| NiP/Fe₃P₂O₅                 | NPs dropcasting             | 2.02                        | 62% at 1.23 V RHE in 3h | this work |
| CoLa(OH)₃                  | electrodeposition            | 2                           | 93% at 0.8 V vs CE in 1.1h | [32] |
| Ru-complex                  | dip-coating                 | 1.4                         | ~40% at 1.23 V RHE in 1.8h | [33] |
| CoPi                        | electrodeposition            | 1.4                         | ~80% at 1.23 V RHE in 1.8h | [33] |
| CoFe-PB                     | immersion                   | 1.1                         | ~45% at 1.23 V RHE in 15 h | [34] |

⁻PED = Photo-assisted electrodeposition

⁻CBD = Chemical bath deposition

Gray rows: BiVO₄ is fully covered with robust cocatalyst layer

White rows: BiVO₄ is partially covered with nanosized particle or monolayer of molecule
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