Electronic Supplementary Material (ESI) for Journal of Materials Chemistry C.


# Supplementary Data

## Efficient and Recyclable $Ni_2P$-$Co_2P/ZrO_2/C$ Nanofiber Photocatalyst for Conversion of Plastic Waste into $H_2$ and Valuable Chemicals

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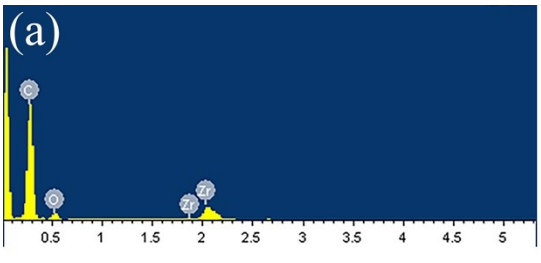
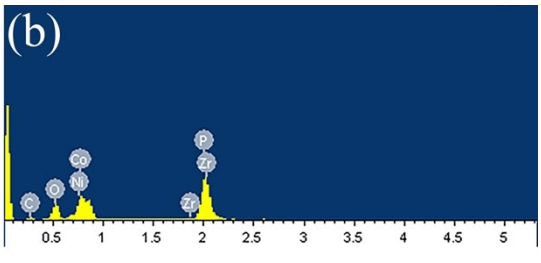



| Element | C | O | Zr |
|---|---|---|---|
| Atomic (%) | 90.99 | 6.86 | 2.15 |
| Weight (%) | 78.13 | 7.85 | 14.02 |




| Element | C | O | Zr | Co | Ni | P |
|---|---|---|---|---|---|---|
| Atomic (%) | 14.64 | 22.77 | 0.94 | 17.60 | 16.77 | 27.28 |
| Weight (%) | 5.21 | 10.78 | 2.54 | 30.69 | 29.13 | 21.65 |

**Fig. S1.** The SEM mode energy-dispersive X-ray (EDX) spectrum : (a) $ZrO_2/C$ NFs and (b) $Ni_2P$-$Co_2P/ZrO_2/C$ NFs.

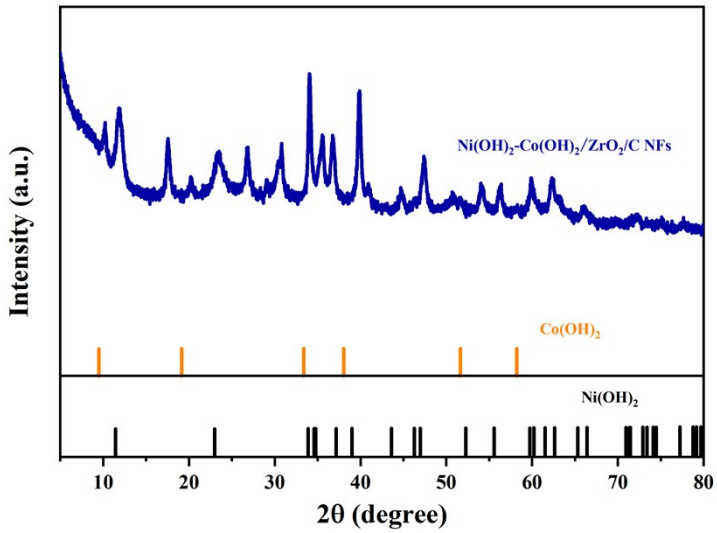



**Fig. S2.** XRD patterns of $Ni(OH)_2$-$CoOH)_2/ZrO_2$/C NFs.

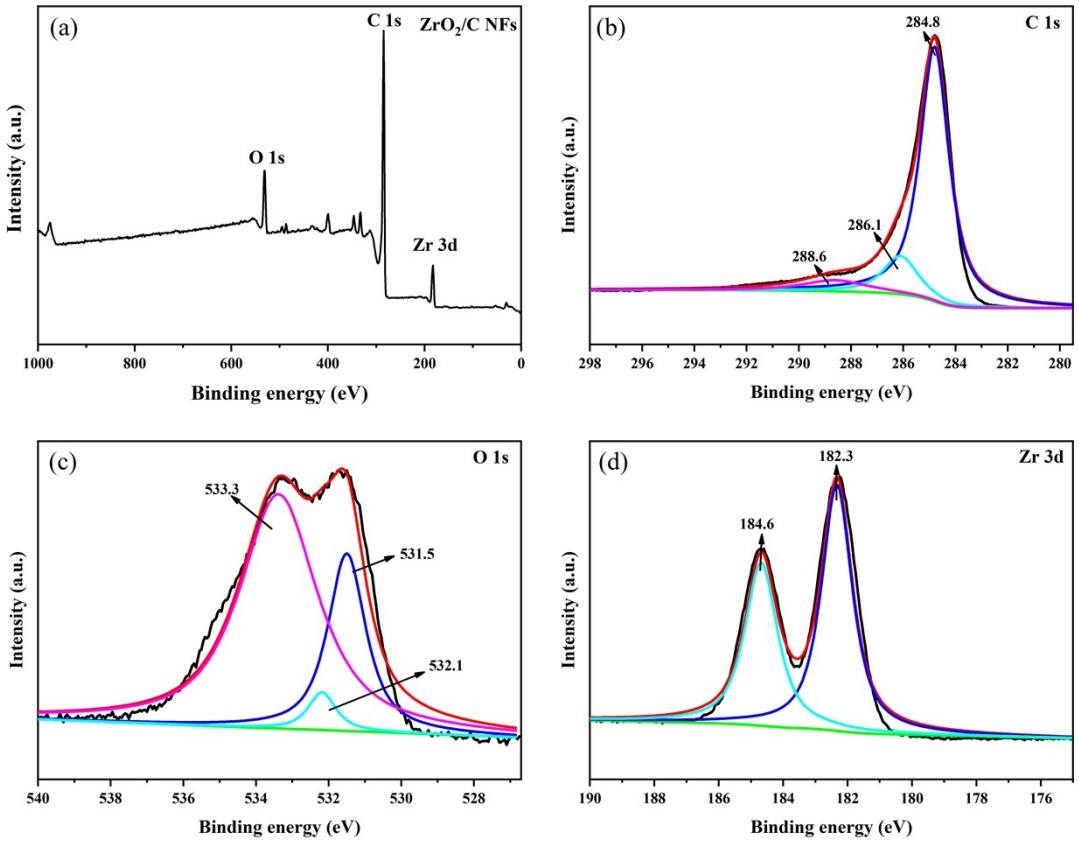



**Fig. S3**. XPS characterization of $ZrO_2$/C NFs: (a) survey spectrum, (b) C 1s region, (c) O 1s region, and (d)Zr 3d region.

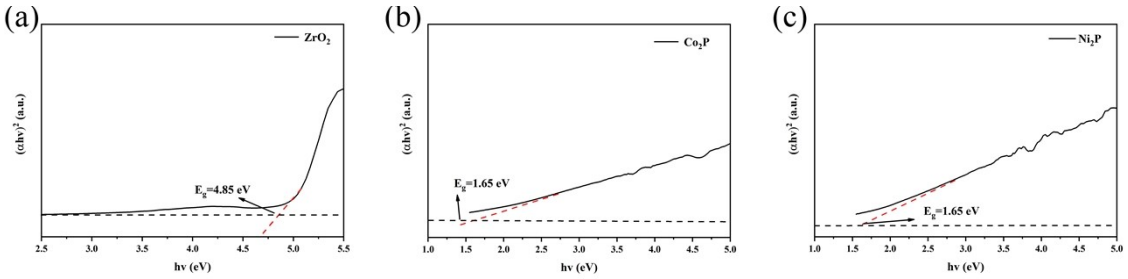



**Fig. S4**. Tauc plots of (a) $ZrO_2$, (b) $Co_2P$ and (c) $Ni_2P$.

The bandgap energy of the synthesized catalyst is obtained using the Tauc (Eq. (1)) [1]:

$$\alpha hv = A\left(hv - E_g\right)^{n/2} \quad (1)$$

wherein "α" signifies the absorption coefficient, "hv" denotes photon energy, and "n" is dependent on the transition type of catalyst. Extrapolate the linear portions obtained from the graphs to the horizontal axis, and the intersection points represent the values of the bandgap width.

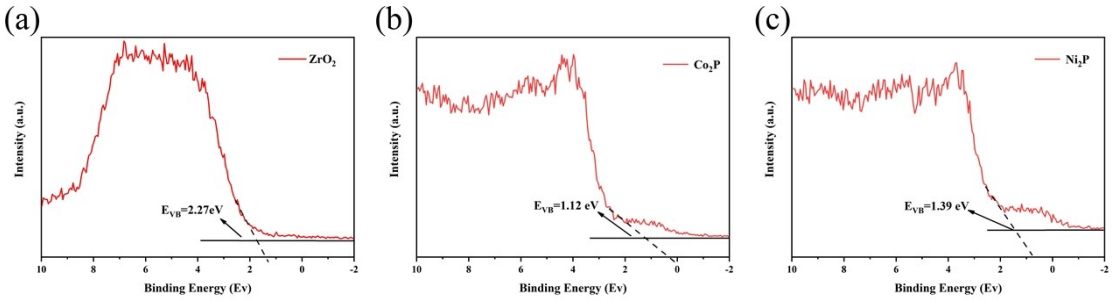



**Fig. S5**. XPS valence-band spectra of (a) $ZrO_2$, (b) $Co_2P$ and (c) $Ni_2P$.

In Fig. S5, based on the VB XPS spectra of $ZrO_2$, $Co_2P$ and $Ni_2P$, linear extensions were observed near 2 eV and 3 eV, and the intersections obtained by extending the horizontal portions below 0 eV correspond to the energy positions of the valence bands of $ZrO_2$, $Co_2P$ and $Ni_2P$, respectively.

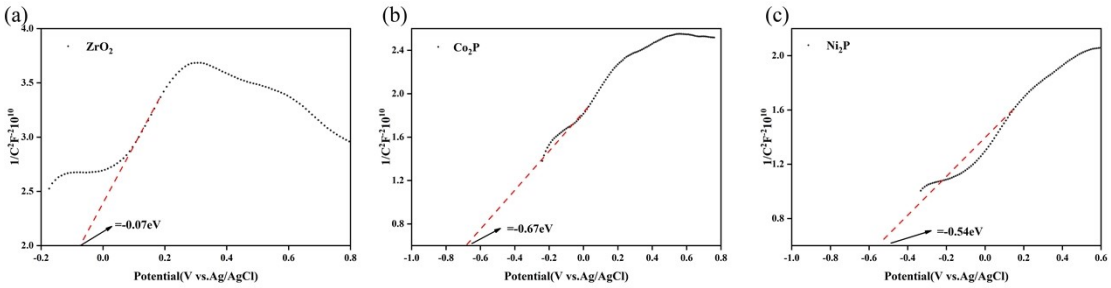



**Fig. S6**. Mott-Schottky plots of (a) $ZrO_2$, (b) $Co_2P$, (c) $Ni_2P$.

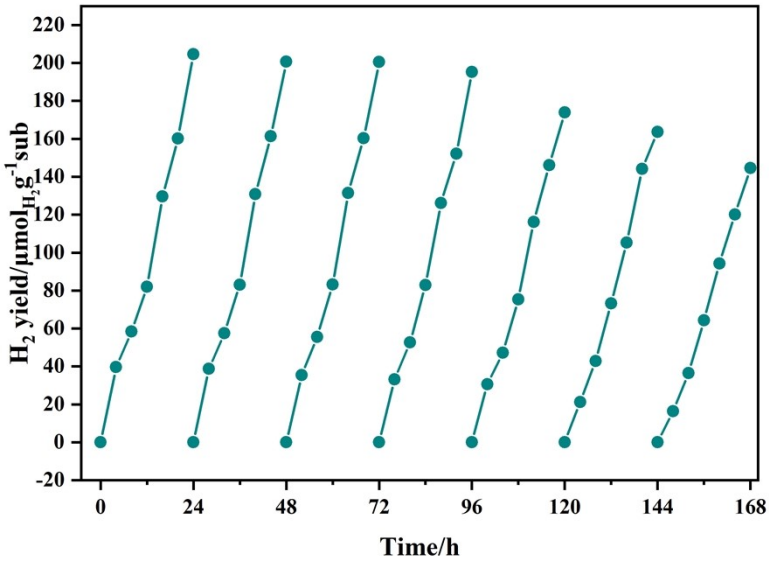



**Fig. S7**. Cyclic catalysis experiments.

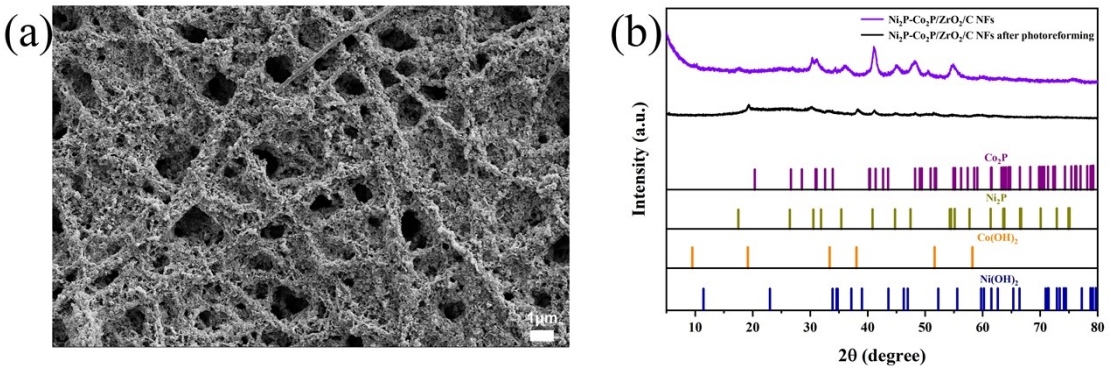



**Fig. S8.** (a) SEM images and (b) XRD patterns of $Ni_2P$-$Co_2P$/$ZrO_2$/C NFs after photoreforming.

**Table S1.** Control experiments for photoreforming of polymers over $Ni_2P$-$Co_2P$/$ZrO_2$/C NFs

Conditions (unless stated otherwise below): pre-treated polymer (50 mg), aqueous KOH (10 M, 10 mL), sealed photoreactor, 500 W mercury lamp. Yields are cumulative values. σ is the standard deviation calculated from 4 samples.

| **Experiment Details** | **Time (h)** | **Yield ± σ** ($\mu mol_{H_2} gsub^{-1}$) |
|---|---|---|
| No $Ni_2P$-$Co_2P$ | 0 | 0±0 |
| | 12 | 13.67±1.36 |
| | 24 | 34.47±2.04 |
| No $Ni_2P$ | 0 | 0±0 |
| | 12 | 42.04±7.34 |
| | 24 | 134.38±11.56 |
| No $Co_2P$ | 0 | 0±0 |
| | 12 | 58.24±8.01 |
| | 24 | 144.55±5.79 |
| No substrate | 0 | 0±0 |
| | 12 | 0±0 |
| | 24 | 0±0 |
| No light | 0 | 0±0 |
| | 12 | 0±0 |
| | 24 | 0±0 |

| | | |
|---|---|---|
| No catalyst | 0 | 0±0 |
| | 12 | 0±0 |
| | 24 | 0±0 |
| No KOH | 0 | 0±0 |
| | 12 | 0±0 |
| | 24 | 0±0 |

**Table S2.** Hydrogen production rates under different alkali concentrations. Conditions (unless stated otherwise below): pre-treated polymer (50 mg), aqueous KOH (2M, 5M, 10 M, 10 mL), sealed photoreactor, 500 W mercury lamp. Yields are cumulative values. σ is the standard deviation calculated from 4 samples.

| **Experiment Details** | **Time (h)** | **Yield ± σ** ($\mu mol_{H_2} gsub^{-1}$) |
|---|---|---|
| $H_2$ yield in 2 M KOH | 0 | 0±0 |
| | 4 | 0±0 |
| | 8 | 6.58±0.52 |
| | 12 | 11.13±0.78 |
| | 16 | 15.67±1.24 |
| | 20 | 26.19±2.03 |
| | 24 | 35.27±3.93 |

| | | |
|---|---|---|
| $H_2$ yield in 5 M KOH | 0 | 0±0 |
| | 4 | 8.74±0.54 |
| | 8 | 17.52±2.83 |
| | 12 | 38.09±2.96 |
| | 16 | 61.99±2.88 |
| | 20 | 75.60±9.30 |
| | 24 | 98.04±5.52 |
| $H_2$ yield in 10 M KOH | 0 | 0±0 |
| | 4 | 44.78±2.18 |
| | 8 | 64.58±3.02 |
| | 12 | 95.83±3.14 |
| | 16 | 125.14±3.22 |
| | 20 | 159.98±5.92 |
| | 24 | 207.56±5.92 |

**Table S3.** Photocatalytic PET hydrolysis products. Conditions (unless stated otherwise below) : pre-treated substrate (50 mg), aqueous KOH (10 M, 10 mL), sealed photoreactor, 500 W mercury lamp. Yields are cumulative values. σ is the standard deviation calculated from 4 samples.

| **Substrate** | **Time (h)** | **Yield ± σ** ($\mu mol_{H_2} gsub^{-1}$) |
|---|---|---|
| Ethylene glycol | 0 | 0±0 |

| | | |
|---|---|---|
| | 12 | 47.28±6.72 |
| | 24 | 69.32±4.57 |
| Terephthalate | 0 | 0±0 |
| | 12 | 0±0 |
| | 24 | 0±0 |

**Table S4.** Long-term photoreforming of real-world waste. Conditions (unless stated otherwise bel ow) : pre-treated polymer (50 mg), aqueous KOH (10 M, 10 mL), sealed photoreactor, 500 W mer cury lamp. Yields are cumulative values. σ is the standard deviation calculated from 4 samples.

| **Experiment Details** | **Time (h)** | **Yield ± σ** ($\mu mol_{H_2} gsub^{-1}$) |
|---|---|---|
| **photoreforming of pretreated PET fibers** | 0 | 0±0 |
| | 12 | 84.61±9.21 |
| | 24 | 148.82±6.96 |
| | 36 | 230.17±7.32 |
| | 48 | 324.18±5.21 |
| | 60 | 458.21±3.33 |
| | 72 | 577.86± 8.09 |
| | 84 | 660.12± 2.68 |
| | 96 | 708.06±8.65 |

| | | |
|---|---|---|
| **photoreforming of pretreated PET particles** | 0 | 0±0 |
| | 12 | 120.33±6.11 |
| | 24 | 195.63±12.58 |
| | 36 | 298.31±8.41 |
| | 48 | 396.09±10.73 |
| | 60 | 499.66±10.18 |
| | 72 | 623.49± 7.11 |
| | 84 | 670.67± 7.85 |
| | 96 | 719.45±9.68 |
| **photoreforming of pretreated PET cups** | 0 | 0±0 |
| | 12 | 161.24±7.93 |
| | 24 | 266.05±10.78 |
| | 36 | 378.27±7.86 |
| | 48 | 504.49±13.16 |
| | 60 | 606.71±12.44 |
| | 72 | 689.99± 14.06 |
| | 84 | 768.93± 3.35 |
| | 96 | 822.13±6.88 |

1. F. Di Quarto, C. Sunseri, S. Piazza and M. Romano, *J. Phys. Chem. B*, 1997, **101**, 333-340.