Supporting Information

Photoinduced Charge Transfer Dynamics in WO₃/BiVO₄ Photoanodes Probed through Mid-IR Transient Absorption Spectroscopy

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1. Experimental

1.1. Materials

The following chemicals were employed in the present work: tungsten(VI) ethoxide 99.8% (5% w/v in ethanol), ammonium vanadium oxide, bismuth(III) nitrate pentahydrate ACS 98%, benzyl alcohol ACS 99% (Alpha Aesar), ethyl cellulose (MP Biomedics), poly(vinyl alcohol) >99%, citric acid 99%, FeSO₄ 98% (Aldrich), glacial acetic acid, anhydrous sodium sulfate, and sodium sulphite (Fisher Scientific).

1.2. Photoelectrodes preparation

WO₃ films were prepared according to the already described procedure,¹ with minor changes. Briefly, 1.0 mL of tungsten ethoxide, 5 wt% in ethanol, was added to 42 mg of citric acid under nitrogen atmosphere inside a glovebox. Once citric acid was completely dissolved, benzyl alcohol (0.3 mL) and ethyl cellulose (40 mg) were added to the solution, which was stirred overnight at 70 °C to attain the complete dissolution of ethyl cellulose. The so-obtained paste, containing 0.085 M tungsten, is stable for several weeks. 100 µL of the paste were deposited on a 2.5 ⁻ 2.5 cm² fluorine-doped tin oxide (FTO) glass electrode, by spin coating at 500 rpm s⁻¹ up to 1000 rpm, then at 1000 rpm s⁻¹ up to 3000 rpm and finally at 3000 rpm s⁻¹ up to 6000 rpm, which was maintained for 30 s. Prior to deposition, the FTO glass was cleaned by 15 min-long sonication, first in an aqueous soap solution, then in ethanol and finally in water. After being coated, the so-obtained film was dried for 10 min at 70 °C and then calcined on a hot plate at 400 °C for 10 min. Then it was removed from the hotplate and cooled to room temperature. This coating procedure was repeated nine times. Finally, the film was annealed for 1 h at 500 °C. These WO₃ films were 410 nm thick, as measured with a Bruker DektaXT profilometer.

The BiVO₄ and the combined WO₃/BiVO₄ photoanodes were prepared by directly coating FTO or WO₃ electrodes, respectively, with BiVO₄ films prepared according to a procedure similar to that reported elsewhere.² Typically, 0.002 mol of Bi(NO₃)₃ and NH₄VO₃ were added to 6 mL of 23.3% HNO₃ containing 0.004 mol of citric acid. A dense paste was obtained by adding 0.04 g of poly(vinyl alcohol) and 0.25 mL of acetic acid to 1.0 mL of the above solution and the mixture was stirred up to complete dissolution of the precursor. A 45 nm thick BiVO₄ layer was obtained by spin coating the paste at 4000 rpm for 30

s on FTO or WO₃-coated FTO. The so-obtained film was then dried for 1 h at 80 °C and annealed for 1 h at 500 °C.

BiVO₄ films on CaF₂ were prepared employing an organic solvent based procedure, the water based procedure employed for photoanode preparation being inadequate in the case of the CaF₂ substrate. Stoichiometric amounts of bismuth 2-ethoxyethanoate and vanadyl oxy-isopropoxide were added to an acetyl acetone/isopropanol 50 vol% mixture. The dark red solution was spin coated for 20 s at 200 rpm on a CaF₂ in a glovebox under nitrogen atmosphere, then dried at 70 °C under N₂ and finally calcined for 1 h at 500 °C in air. In order to avoid any thermal shock of the so prepared /BiVO₄ on CaF₂ films (CaF₂ is more fragile than FTO), they were then let cool down to below 250 °C before being removed from the hotplate.

Multilayered WO₃ films on CaF₂ were prepared with the same procedure used for the preparation of WO₃ electrodes on FTO, with only minor changes necessary to prevent CaF₂ thermal damage. After the 10 min-long drying process, the WO₃ film was slowly heated to 400 °C on the hotplate. After 30 min, heating was turned off and the CaF₂/WO₃ film was removed from the hotplate only after its temperature was below 250 °C. The spin coating, drying and calcination procedure was repeated nine times. The CaF₂/WO₃/BiVO₄ film was prepared by coating with BiVO₄ a WO₃ film deposited on CaF₂ by the same procedure employed for photoanode preparation.

1.3. Photoelectrochemical characterization

Photoelectrochemical (PEC) measurements were carried out using a three electrode cell with an Ag/AgCl (3.0 M NaCl) reference electrode, a platinum gauze as a counter electrode and a Princeton Applied Research 2263 (PARstat) potentiostat. The photoanodes were tested under back side illumination (through the FTO/BiVO₄ or FTO/WO₃/BiVO₄ interface), in contact with a 0.5 M Na₂SO₄ aqueous solution at pH = 7. The light source was an Oriel, Model 81172 solar simulator providing AM 1.5G simulated solar illumination with 100 mW cm⁻² intensity (1 sun). Both semiconductor oxides are stable at neutral pH and no noticeable degradation was observed under irradiation. The potential *vs*.

Ag/AgCl was converted into the RHE scale using the following equation: $E_{RHE} = E_{AgCl} + 0.059 \text{ pH} + E_{AgCl}^{\circ}$, with E_{AgCl}° (3.0 M NaCl) = 0.210 V at 25 °C.

IPCE (Incident Photon to Current Efficiency) measurements were carried out in Na₂SO₄ 0.5 M (pH 7), using the same single-compartment three-electrode cell used in photoelectrochemical experiments. The IPCE was calculated using the following equation:

$$IPCE = \frac{[1240 \times J]}{P_{\lambda} \times \lambda} \times 100$$

where *J* is the photocurrent density (mA cm⁻²) and P_1 (mW cm⁻²) is the power of the monochromatic light at wavelength I (nm).³ The IPCE enhancement is defined as follows (eq 1):

$$IPCE_{enhancement} (\%) = IPCE_{WO_3/BiVO_4} - (IPCE_{WO_3} + IPCE_{BiVO_4})$$
(1)

1.4. IR spectroscopy of photoreduced WO₃

WO₃ photoreduction was performed by irradiating a WO₃ film prepared on CaF₂ in deaerated ethanol. The WO₃ film was immersed in ethanol, sealed in a closed vessel under nitrogen in a glovebox and reduced under simulated solar irradiation with 150 mW cm⁻². After 2 h-long irradiation the vessel was opened and the deep blue film was quickly removed from the ethanol solution, washed with fresh ethanol, dried and moved to a PerkinElmer Spectrum Two FTIR spectrophotometer. The relaxation of reduced WO₃ was tracked by recording the IR spectra at different time intervals in the following 1.5 hlong exposure to air. The difference absorbance spectra were obtained by subtracting the spectrum of the fully recovered film from the spectra recorded at different time.

1.5. IR spectroscopy of chemically reduced BiVO₄

A calcined BiVO₄ film on a CaF₂ disk was reduced in an aqueous 0.1 M NaBH₄ solution. After 1 minlong immersion, the reduced film was quickly washed with distilled water and then moved to a PerkinElmer Spectrum Two FTIR spectrophotometer. The relaxation of reduced BiVO₄ was tracked by recording the IR spectra at different time intervals in the following 6 h-long exposure to air. The difference absorbance spectra were obtained by subtracting the spectrum of the fully recovered film from the spectra recorded at different time.

1.6. IR Transient absorption spectroscopy and flash photolysis

1.6.1 Ultrafast transient mid-IR absorption spectroscopy

The output of a Ti:sapphire amplifier with integrated oscillator and pump lasers (800 nm, 40 fs, 3 kHz, Libra LHE, Coherent Inc.) was split into two beams which were used to pump two TOPAS Primes coupled with frequency mixers (Light Conversion Ltd). This produced a depolarized visible pump pulse (410 nm or 500 nm) and a broad mid-IR probe spectrum, which were used for a femtosecond transient absorption spectrometer (Helios IR, Ultrafast Systems LLC). Pump pulse energies were adjusted using a neutral density filter placed prior the sample, and was varied from 10 nJ – 300 nJ in intensity dependent experiments. The spot sizes were ~2500 μ m² for 410 nm pump and ~ 1800 μ m² for 500 nm pump. Prior to reaching the sample, the probe beam was split into equal intensity probe and reference beams. All films were prepared on CaF₂ windows and measured in a liquid cell (Specac). The detection of probe and reference beams was done using a femtosecond transient absorption spectrometer (Helios IR, Ultrafast Systems LLC). The instrument response function for the experiments was approximately 200 fs. During the measurements, the samples were moved manually to minimize possible laser-induced sample damage.

1.6.2 Nano-second transient mid-IR absorption spectroscopy

The employed setup was used in previous work.⁴ A frequency doubled Q-switched Nd:YAG laser (Quanta-Ray ProSeries, Spectra-Physics) was employed to obtain a 355 nm pump light with a FWHM of 10 ns. The 355 nm pump light was also used through a MOPO crystal to generate 500 nm pump light. The pump energy at the sample was 10 mJ/pulse. Probing was done with the continuous wave quantum cascade (QC) IR laser with a tuning capability between 1960 and 2150 cm⁻¹ (Daylight Solutions). For IR detection a liquid nitrogen-cooled mercury-cadmium-telluride (MCT) detector (KMPV10-1-J2, Kolmar Technologies, Inc,) was used. The IR probe light was overlapped with the pump beam in a quasi-co-

linear arrangement at 25° angle. Transient absorption traces were acquired with a Tektronix TDS 3052 500 MHz (5GS/s) oscilloscope in connection with the L900 software (Edinburgh Instruments) and processed using Origin 9 software.

1.6.3 Fitting equations

The signal decay recorded with $BiVO_4$ films were fitted according to eq 2, while the traces recorded with WO_3 films and with the coupled system were fitted according to eq 3.

$$\Delta A_{decay} = A_1 e^{-t/\tau_1} + A_2 e^{-t/\tau_2} + \Delta A_0$$
(2)

$$\Delta A_{decay} = A_1 e^{-t/\tau_1} + A_2 e^{-t/\tau_2} + A_r e^{-t/\tau_3} + \Delta A_0$$
(3)

where τ_1 , τ_2 and τ_3 are the lifetimes of the decay processes and A_1 , A_2 and A_3 are the weighted coefficients representing the contribution of each process to the overall decay. ΔA_0 is the offset (set as zero in the fitting).

2. Characterization

2.1. Mid-IR spectra of reduced WO₃

The photoreduction of a WO₃ film was performed in deaerated ethanol, as described in section 1.4. As in the case of colloidal TiO₂ and WO₃ and of other photochromic materials,^{5–7} under such conditions photogenerated holes are filled with the electrons provided by ethanol and photopromoted electrons can be trapped as W⁵⁺ at intra band gap sites, originating an absorption signal extending from the visible to the near infrared region.^{8,9} This signal progressively disappeared when the WO₃ film was in contact with air, within the IR spectrophotometer. The difference spectra, obtained by subtracting the spectrum of the fully recovered film (after 90 min-long exposure to air) from the absorption spectra recorded at different times, are shown in **Figure S1**.



Figure S1. Difference absorption spectra recorded during the recovery of a reduced WO₃ film in contact with air. Difference spectra were obtained by subtracting the spectrum of the fully recovered film (recorded after 90 min-long exposure to air) from the spectra recorded at different times.

2.2. IR traces of reduced BiVO₄ in the mid-IR range



Figure S2. Normalized difference absorbance signals recorded at 2050 cm⁻¹ and at 3350 cm⁻¹ during the mid-IR spectrum decay recorded with reduced $BiVO_4$ in contact with air. The similarity of the decay traces indicates that the broad absorption feature in the mid-IR is originated by one species only.

2.3. UV-Vis absorption spectra



Figure S3. Absorption spectra of the WO₃, BiVO₄ and WO₃/BiVO₄ films on FTO. All WO₃ films were 410 nm thick, all BiVO₄ films were 45 nm thick.

2.4. IPCE measurements



Figure S4. Left panel: IPCE measurements at 1.23 V vs. RHE recorded with the electrodes in contact with a 0.5 M Na₂SO₄ aqueous solution. The IPCE enhancement factor defined by **eq 1** is also shown. Right panel: corresponding internal quantum efficiency (IQE) analysis obtained by normalizing the IPCE values with the absorbed light.

Figure S4 shows the incident photon to current efficiency (IPCE) and the internal quantum efficiency (IQE) of the WO₃ and BiVO₄ films and of the composite heterojunction recorded at 1.23 V vs. RHE. WO₃

and BiVO₄ electrodes exhibit a photocurrent onset at 460 and 520 nm, respectively, in agreement with their corresponding band gap energy. Although BiVO₄ absorbs a larger fraction of visible light, its conversion efficiency is lower compared to that of WO₃, due to its intrinsic poor charge separation efficiency and low interfacial charge-transfer capability across the solid-liquid junction.^{10,11} However, the efficient electron-hole separation occurring in the heterojunction system clearly boosts BiVO₄ photoactivity. The IPCE enhancement factor (black line in **Figure S4**), defined as the difference between the IPCE obtained with the coupled system and the sum of the IPCE values obtained with the two individual photoanodes (see **eq 1**), is a useful tool to highlight the wavelength dependent behavior observed in the WO₃/BiVO₄ heterojunction.¹² Indeed, the IPCE enhancement factor is positive at wavelengths longer than the WO₃ absorption onset (ca. 460 nm). In contrast, for wavelengths shorter than 400 nm, the IPCE enhancement is negative, i.e. the IPCE of the heterojunction system falls below the sum of the IPCE curves measured with the two individual semiconductors.¹² This indicates that charge recombination of electrons photopromoted in WO₃ with BiVO₄ holes occur upon simultaneous BiVO₄ and WO₃ excitation.



2.5. Femtosecond IR TA spectra

Figure S5. fs-IR TA spectra recorded with the WO₃/BiVO₄ electrode at different delay times after excitation at (A) 410 nm and (B) 500 nm.



2.6. Power dependence in BiVO₄ upon 410 nm excitation

Figure S6. (A) Power dependence of the TA IR signals recorded at 2050 cm⁻¹ with the BiVO₄ film upon excitation at 410 nm in the 30-300 μ W range. (B) Fitting of the normalized absorption difference traces.

The pump power was varied from 30 μ W, the lowest value allowing to obtain a measurable Δ A signal, up to 300 μ W. The resulting difference absorption Δ A traces are reported in **Figure S6A**. The Δ A magnitude shows a nonlinear increase with increasing pump power, with the signal recorded at 300 μ W being just 1.4 times larger than the signal recorded at 150 μ W (Δ A = 0.71 × 10⁻³ with respect to Δ A = 0.5 × 10⁻³, respectively). This is possibly due to a progressive acceleration of the initial fast decay process which may become faster than the time resolution of the employed femtosecond setup, rather than to CB saturation. On the other hand, in previous work¹² we found that trapped holes monitored in the visible region show a different behavior in excitation power dependence experiments, with Δ A linearly increasing with increasing pump power. These two facts, together with the observation (see the main text) that the transient hole and electron signals, monitored at 470 nm and 2050 cm⁻¹ respectively, decay in different time scales, indicate that the two signals appear to be uncorrelated. That is, the relaxation of the photoexcited electrons monitored through the IR signal does not necessarily result in trapped holes signal decay and therefore does not directly lead to electron-hole (e⁻/h⁺⁾ recombination. This in

turn supports the hypothesis that photoexcited electrons monitored at 2050 cm⁻¹ may relax to IR unobservable states.¹³

The normalized IR traces are reported in **Figure S6B**. All traces instantaneously maximize within the response function of the experimental setup except for that recorded at the lowest power which maximizes ca. 3 ps after photoexcitation, probably due to the high noise to signal ratio at this excitation power. For this reason we did not consider the dataset obtained by the lowest power pump. The ΔA decays were fitted according to **eq 2** and the fitting traces are reported in **Figure S6B**. With increasing the pump power the overall decay become progressively faster and both time constants show a threefold decrease with respect to the ΔA decay under 75 µW excitation, when a 300 µW excitation was employed

(Table S1).

Furthermore, by comparing these results with the power dependence of trapped holes reported in **ref. 12**, we observe that the long time component T_2 (in the ns range) of the trapped hole signal decay is unaffected by the pump power, which again supports the hypothesis that the fast decay observed for free electrons monitored in the IR may not necessarily lead to recombination. However, the pump power does affect the fast decay component T_1 (15-30 ps) of the trapped holes decay and shows a 40% decrease by increasing six times the pump power.

	Power			
	30 µW	75 µW	150 µW	300 µW
A ₁ (%)	42 ± 23	66 ± 7	59 ± 6	61 ± 4
т ₁ (ps)	16 ± 7	18 ± 2	8.6 ±0.9	5.1 ± 0.4
A ₂ (%)	58 ± 17	34 ± 14	41 ± 8	39 ± 7
т ₂ (ps)	170 ± 40	223 ± 49	103 ± 13	75 ± 8
Red. c ²	6.6 10 ⁻³	2.7 10 ⁻³	1.4 10 ⁻³	1.05 10 ⁻³
Adj. R ²	0.94	0.98	0.989	0.99

Table S1. Fitting parameters of the difference absorption traces obtained upon 410 nm excitation of BiVO₄ with different excitation power (Figure S6A).

Therefore, both the electron IR signal and the fast decay component of the hole signal become faster as the pump power get increased, in agreement with the evidence provided by Ravensberger et al. that the fast decay component (with time constant of 40 ps) is related to hot electrons/trapped holes recombination¹⁴ and with the assignment of the IR signal to free electrons. Thus, the ultrafast decay exhibited by the mid-IR signal can be ascribed to the trapping and cooling of free electrons into IR unobservable states, though a degree of charge recombination cannot be excluded and may be active in the first tenths of picosecond.



2.7. Electron dynamics in BiVO₄ upon 410 and 500 nm excitation

Figure S7. Transient absorption difference ΔA profiles at 2050 cm⁻¹ recorded with a BiVO₄ on CaF₂ upon excitation at 410 and 500 nm (blue triangles and red diamonds, respectively). The pump power was 0.8 and 1.8 mW for the 410 and 500 nm pump, respectively, in order to generate the same number of charge carriers, based on the absorbance of the BiVO₄ film.

The absorption difference ΔA profiles reported in **Figure S7** were obtained by tuning the pump power at the employed pump wavelengths, in order to photogenerate the same amount of charge carriers, thus

avoiding any possible difference in recombination dynamics due to a different density of photoproduced charge carriers.

As shown in **Scheme S1**, when the photon energy was varied from 410 nm (ca. 3 eV) to 500 nm (ca 2.5 eV) the photopromoted electrons had different energy excess (0.6 and 0.1 eV, respectively) above the BiVO₄ CB bottom. Therefore, any difference in the IR signal decay may be related to differences in the relaxation pathways of photopromoted electrons, rather than to processes (common to both excitation conditions) involving the recombination of thermalized CB electrons with VB holes or mediated by intraband gap trap states.



Scheme S1. Excitation energy excess in the BiVO₄ CB of electrons photopromoted with 410 and 500 nm pumps.

	410 nm pump	500 nm pump
A ₁ (%)	70 ± 4	62 ± 4
τ ₁ (ps)	10.0 ± 0.8	2.7 ±0.2
A ₂ (%)	30 ± 9	38 ± 2
τ ₂ (ps)	171 ± 26	43 ± 5
Red. c ²	1.3 10 ⁻³	7.83 10 ⁻⁴
Adj. R ²	0.987	0.99

Table S2. Fitting parameters obtained from the decay traces recorded with BiVO₄ upon excitation at 410 or 500 nm (traces shown in Figure S7)

The decay traces reported in **Figure S7** can be fitted by two exponential decay functions (**eq 2**, see Section **1.6.3**); the so obtained fitting parameters are reported in **Table S2**. The free electrons signal produced upon 500 nm excitation decays faster than the signal produced upon 410 nm excitation. A

faster direct e⁻/h⁺ recombination can be ruled out as the charge carriers population was equal at the two excitation wavelengths. Therefore, the faster decay observed under 500 nm excitation with respect to 410 nm excitation can be ascribed to the lower energy excess which photopromoted free electrons need to lose in order to guench to IR unobservable states.





Figure S8. Comparison between the decay traces of trapped holes and free electrons monitored at 470 nm (continuous lines) and at 2050 cm⁻¹ (dash-dot lines), respectively, recorded with (A) $BiVO_4$ and (B) $WO_3/BiVO_4$ electrodes. The traces recorded in the visible upon $WO_3/BiVO_4$ excitation at 387 and 500 nm are adapted from Grigioni et al.^{1,12}

The dynamics of the two charge carriers upon excitation with ca. 400 nm and 500 nm pumps are compared in **Figure S8** (orange and green traces, respectively) and show that when BiVO₄ in the WO₃/BiVO₄ coupled system is selectively excited (500 nm pump) the lifetime of both photopromoted electrons and VB holes in BiVO₄ is extended with respect the situation in which both oxides are simultaneously excited. This suggests that, by looking through the two spectral (i.e., vis and mid-IR) windows, the effects of charge separation occurring through path A in Scheme 1 (main text) can be monitored in the two photogenerated counterparts.

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