Supporting Information

Activation of Kagome lattice structured Cu$_3$V$_2$O$_7$(OH)$_2$·2H$_2$O volborthite via hydrothermal crystallization for boosting visible light-driven water oxidation

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Reaction setup

**Scheme S1.** Schematic diagram of home-built multizone photocatalytic reaction testing system consisting of eight air-tight quartz reactors and a 500 W mid-pressure Hg lamp. Notably, a relative low-power Hg lamp was irradiated on each reactor. The performance measurements of samples prepared in different batches were reproducibly conducted at least three times.
TEM/HRTEM images

Figure S1. TEM images of the HT volborthite sample (a, b), the auto-correlated HRTEM images of CoO (c) and Co$_3$O$_4$ (d) obtained from the corresponding rectangles in the HRTEM image of 2 wt.% CoO/HT in Figure 3.
Figure S2. (a) Kubelka–Munk transformation of UV-Vis diffuse reflection (UV-Vis DRS) spectra of the volborthites; (b) Tauc plots calculated from the Kubelka-Munk transformation. Top-left inset: photograph for the as-synthesized volborthites taken by an ordinary camera.
High resolution XPS spectra

![Graph showing high resolution XPS spectra of Co 2p on the recycled 2.0 wt% and 10 wt% Co/HT volborthite sample, respectively.]

**Figure S3.** High resolution XPS spectra of Co 2p on the recycled 2.0 wt% and 10 wt% Co/HT volborthite sample, respectively.
**Adsorption capacity and photodegradation pseudo first-order rates**

The adsorption capacity is calculated based on the experimental data and according to the mass balance relationship:

\[
Q_e = \frac{(C_o - C_{eq})V}{m}
\]  

where \(Q_e\) is the amount of dye adsorbed onto the volborthite samples, \(C_o\) and \(C_{eq}\) are the initial and equilibrated dyes concentrations (mg/L), respectively, \(V\) is the volume of solution (L), and \(m\) is the mass of the volborthite samples (g).

The intra-particle diffusion model is expressed by:

\[
Q_e = k_p t^{1/2} + C
\]

where \(k_p\) is the intra-particle diffusion rate constant, \(C\) is a constant related to the thickness of the boundary layer.

The reaction kinetics of photocatalytic degradation is generally followed the pseudo first-order rate model:[3b]

\[- \ln \left( \frac{C_t}{C_0} \right) = kt\]

where \(C_0\) and \(C\) are the concentrations of MB solution at times 0 and \(t\) (mg/L), \(k\) is the photodegradation rate, which is equal to the corresponding slope of the fitting line, and \(t\) is the illumination time (min).
Figure S4. (a) Kinetic adsorption capacity of the as-synthesized volborthite samples and the corresponding related removal efficiencies. (b) Kinetic behavior of intra-particle diffusion model for MB adsorption; (c) The estimated photodegradation pseudo first-order rates for MB adsorption. Error bars represent that all the measurements were reproducibly conducted at least three times with deviations less than 5%.
Photographs for color variations of MB solution

**Figure S5.** Color variations of MB solution over the absorption-desorption and photodegradation processes over HT volborthite sample.
**Figure S6.** XRD pattern of 2.0 wt.% CoO₅-HT volborthite sample after photocatalytic O₂ evolution reaction for 5h.
Theoretical calculation

The conduction band (E_{CB}) and valence band (E_{VB}) positions of Cu_3V_2O_7(OH)_2·2H_2O can be estimated according to Butler and Ginley method using the experimental band gap value and the following equation related to Mulliken electronegativity:\(^1\)

\[
E_{CB} = \chi - E_e - 0.5E_g \quad (1)
\]

\[
E_{CB} = E_{VB} - E_g \quad (2)
\]

where \(\chi\) is the geometric mean value of Mulliken electronegativity of the different atoms of the Cu_3V_2O_7(OH)_2·2H_2O material (~5.98 eV), \(E_e\) is the free electron energy (\(E_e = 4.44 \pm 0.02\) eV) and \(E_g\) is the measured band gap of the Cu_3V_2O_7(OH)_2·2H_2O (\(E_g = ~2.1\) eV). Thus, the corresponding \(E_{CB}\) and \(E_{VB}\) of Cu_3V_2O_7(OH)_2·2H_2O can be calculated to be about +0.49 eV and +2.59 eV, respectively.
Mott-Schottky plots

**Figure S7.** Mott-Schottky plots of the HT volborthite in dark at 5 KHz and 10 KHz and an AC current of 5 mV with a three-electrode system in a 0.5M Na$_2$SO$_4$ solution (pH 6.5).
Comparasion of band gaps and positions

Figure S8. Schematic diagram of the band gaps and positions of the volborthite, BiVO₄, WO₃ and g-C₃N₄.
Reference