Supplementary information

Photocatalytic performance of $Y_2Ti_2O_5S_2$ prepared via carbon disulfide sulfurization

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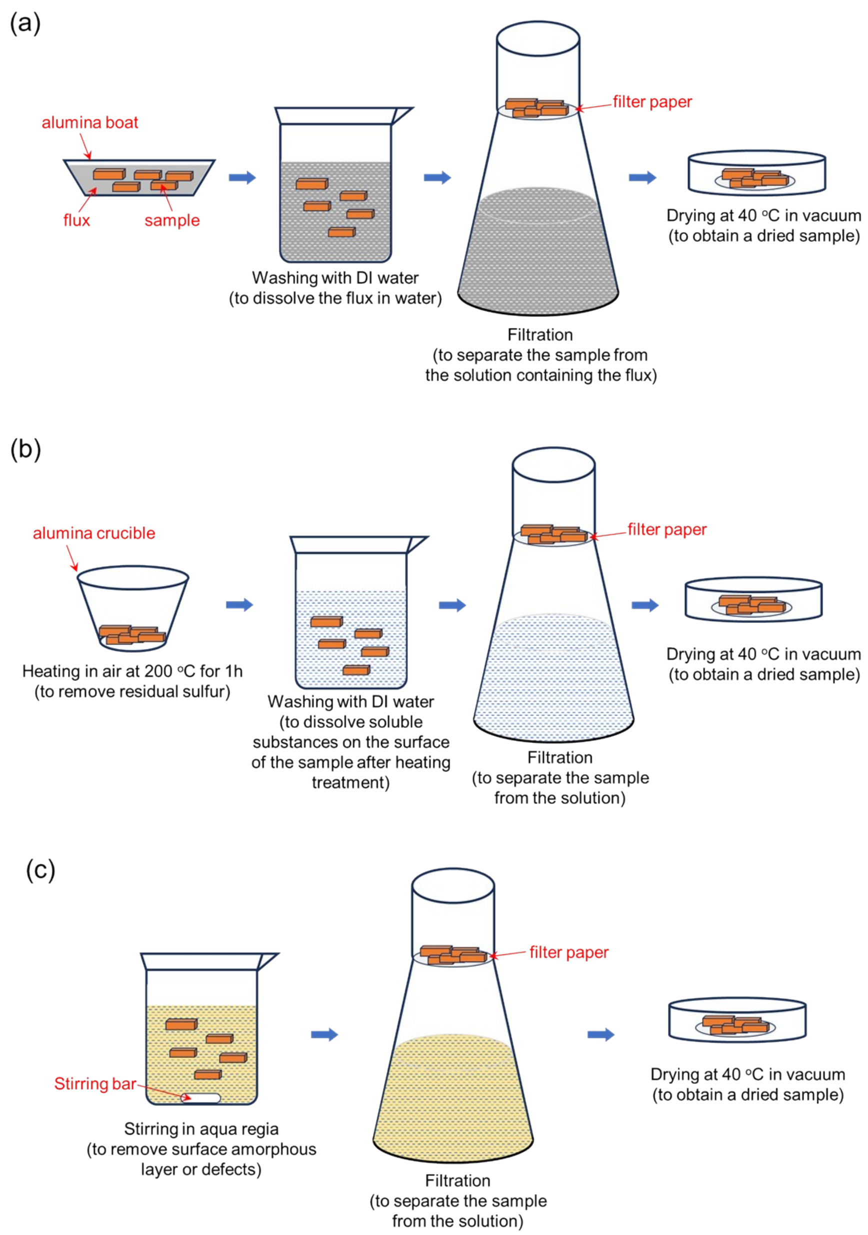

Figure S1. Typical post-treatment procedures for (a) flux removal, (b) residual sulfur removal, and (c) acid treatment.

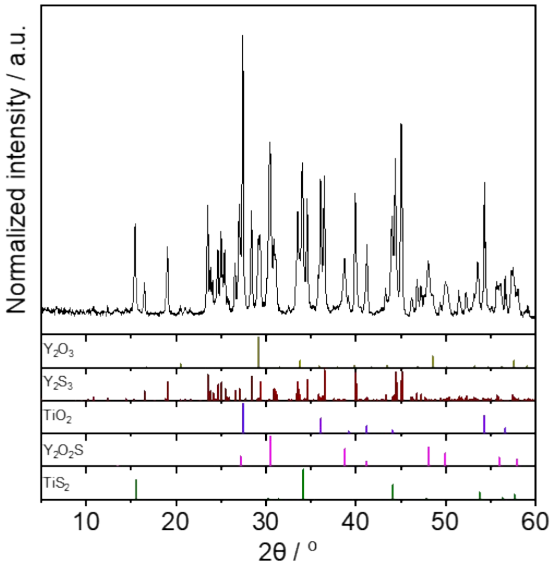

Figure S2. XRD pattern for sample prepared by sulfurization of precursor mixture including Y_2S_3 for 3 h along with patterns for various reference materials.

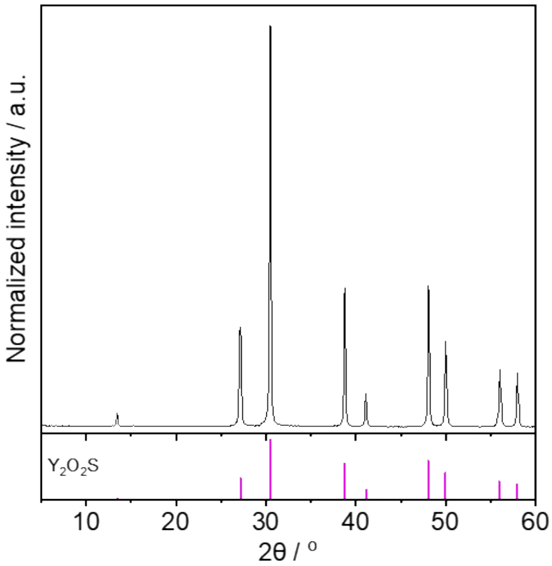

Figure S3. XRD pattern for sample prepared by sulfurization of Y_2O_3 using H_2S at 1150 °C for 3 h along with that for Y_2O_2S as reference. Heating rate: 10 °C/min. H_2S flow rate: 50 mL/min without dilution.

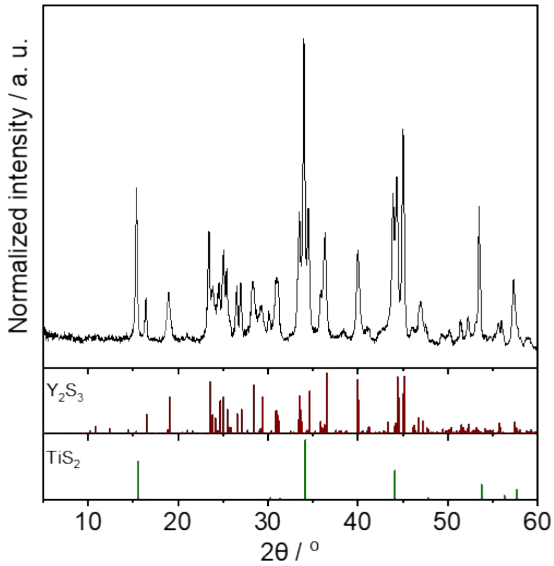

Figure S4. XRD patterns for sample prepared by sulfurization of mixture of Y_2O_3 and TiO_2 for 3 h along with patterns for Y_2S_3 and TiS_2 as references.

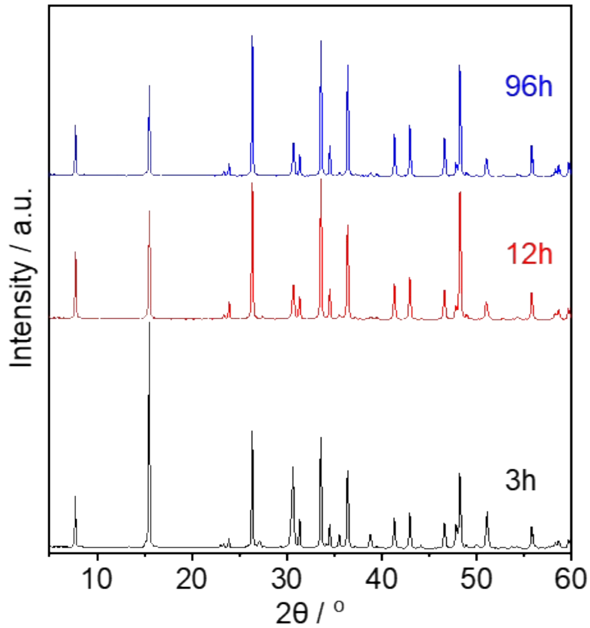

Figure S5. XRD patterns for YTOS samples prepared by SSR method using different heating durations.

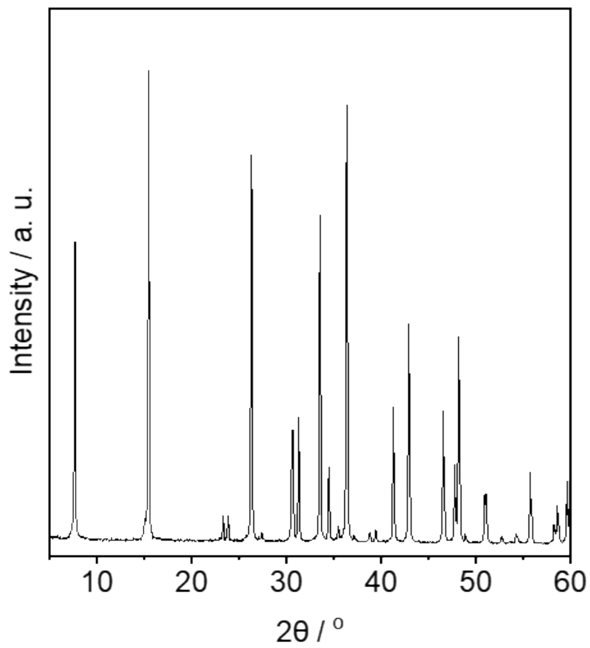

Figure S6. XRD pattern for YTOS-Flux.

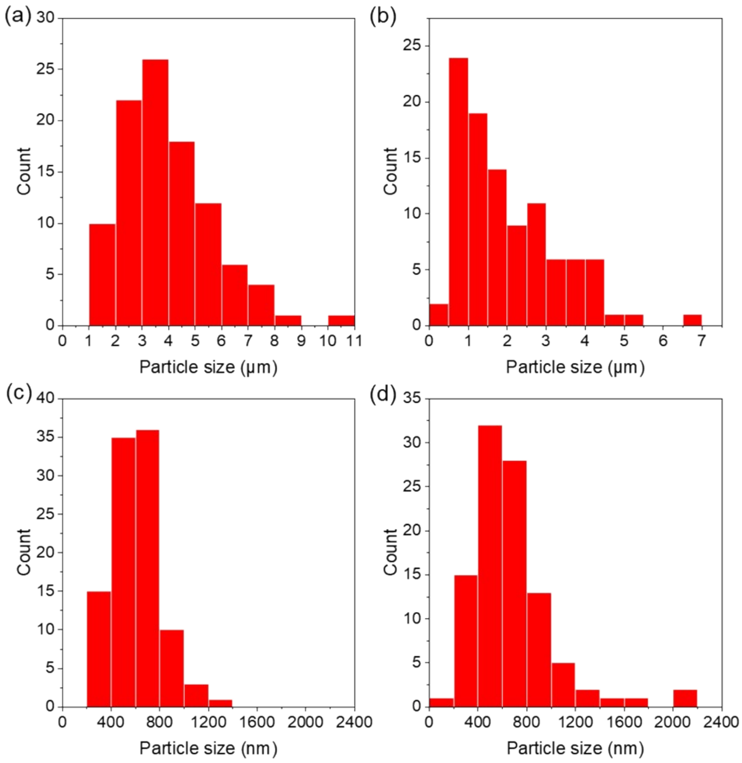

Figure S7. Long axis particle size distributions with 100 particles of (a) YTOS-SSR, (b) YTOS-Flux, (c) YTOS-YOYS, and (d) YTOS-YO samples.

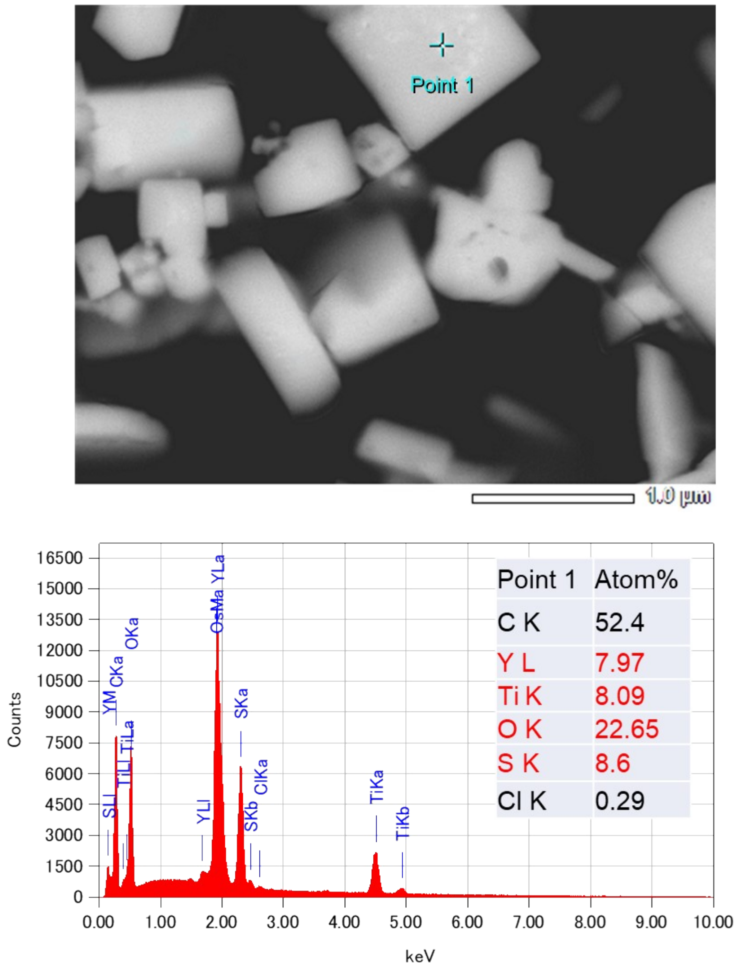

Figure S8. EDS pattern of the YTOS-YO. The results showed the atomic ratio of Y, Ti, O and S was 1.92 : 2 : 5.76 : 2.14, which is close to the stoichiometric ratio of Y, Ti and S in $Y_2Ti_2O_5S_2$. The relatively higher content of oxygen and carbon were ascribed to the embedding of the sample in a resin during the sample preparation for observation.

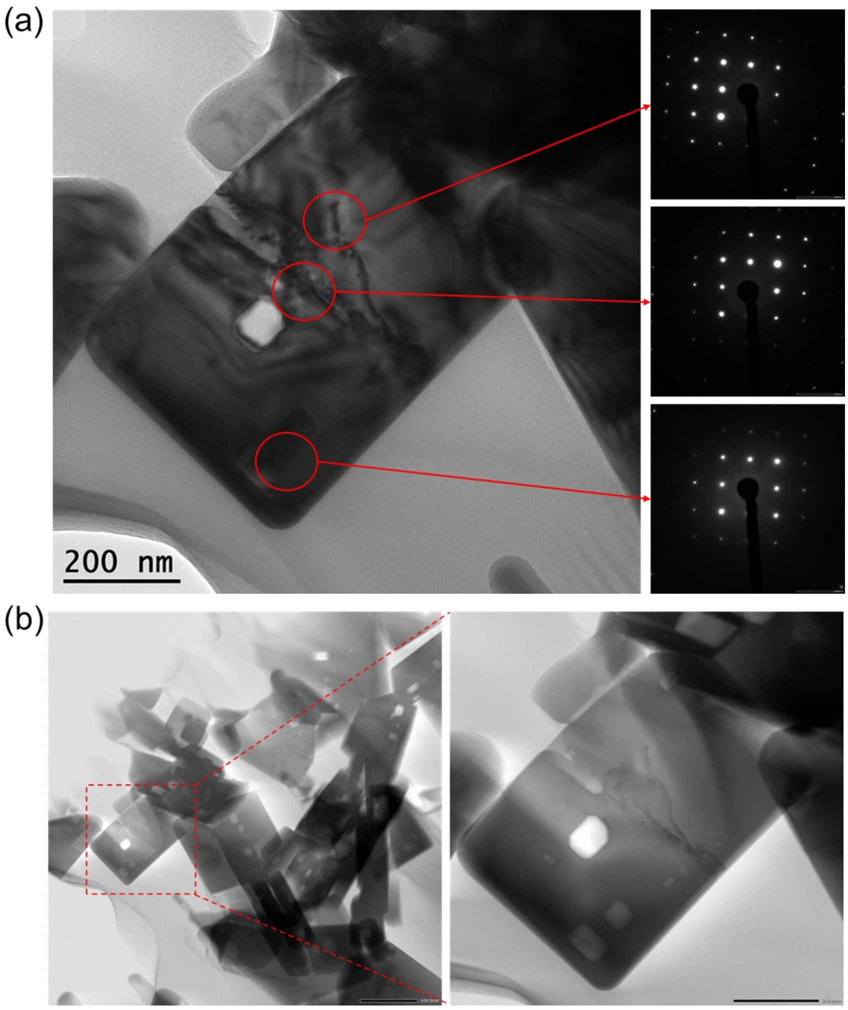

Figure S9. TEM images of (a) basal surface of YTOS-YO specimen and corresponding SAED patterns acquired at different areas, and (b) voids inside YTOS-YO crystals.

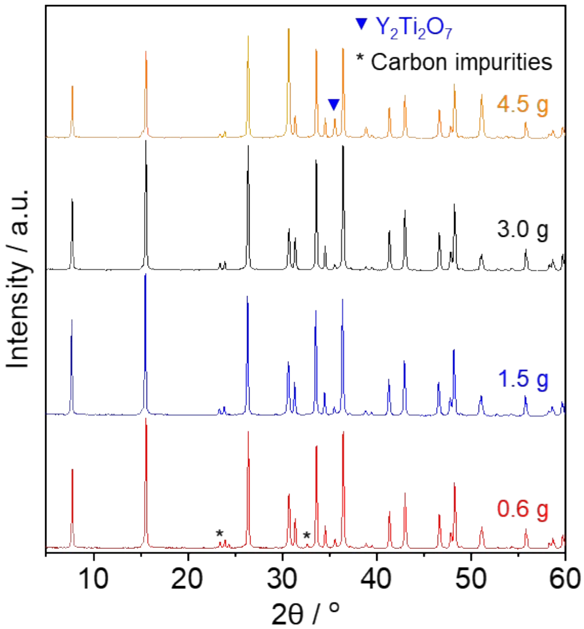

Figure S10. XRD patterns for YTOS-YO specimens synthesized by varying the loading amount of the mixture.

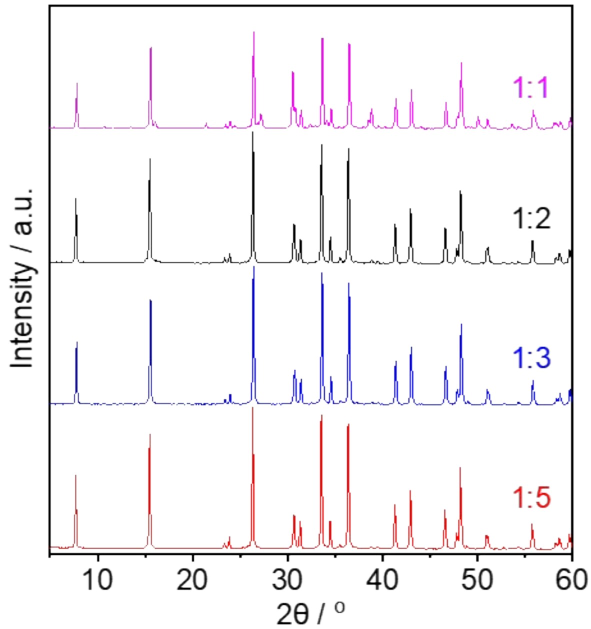

Figure S11. XRD patterns for YTOS-YO specimens synthesized using different precursor-to-flux mass ratios.

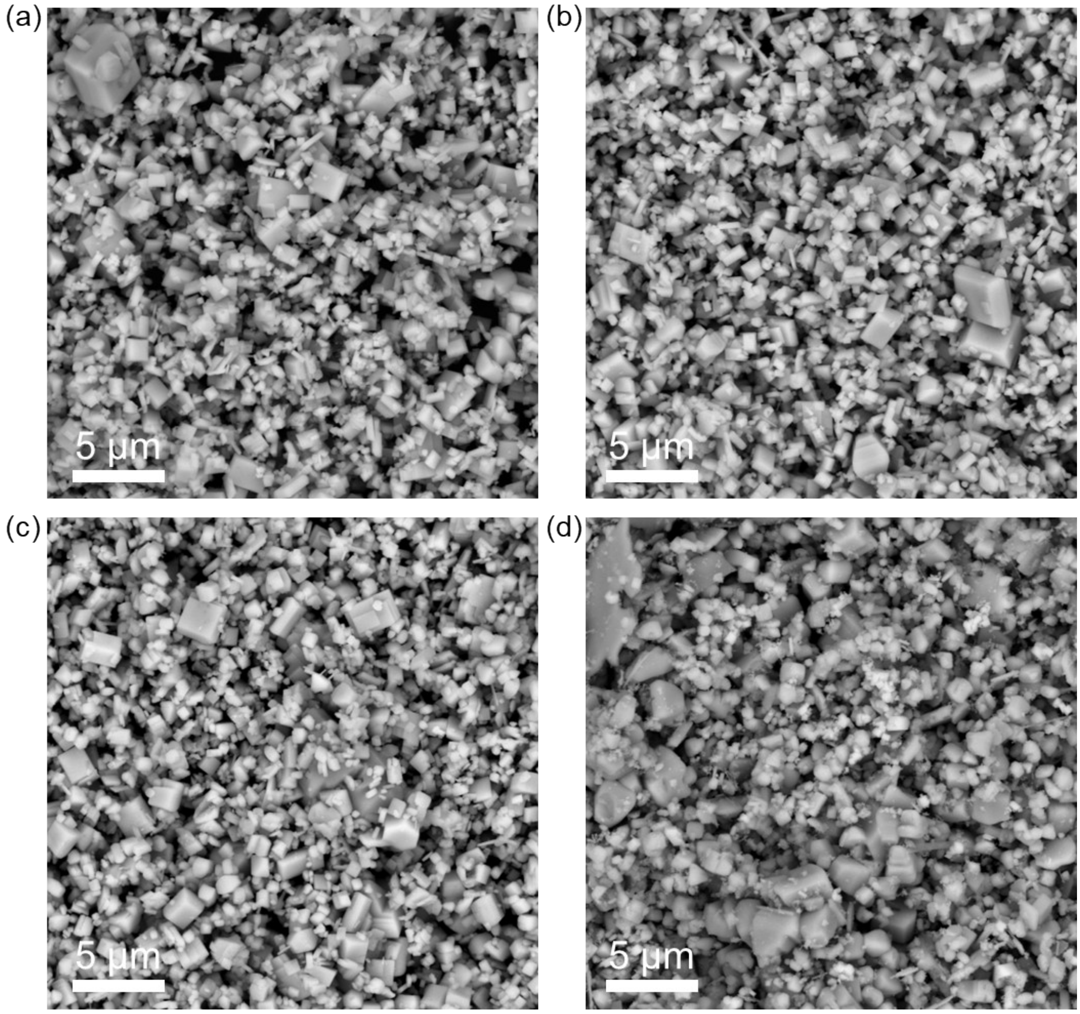

Figure S12. SEM images of YTOS-YO specimens produced using precursor-to-flux mass ratio of (a) 1:5, (b) 1:3, (c) 1:2, and (d) 1:1.

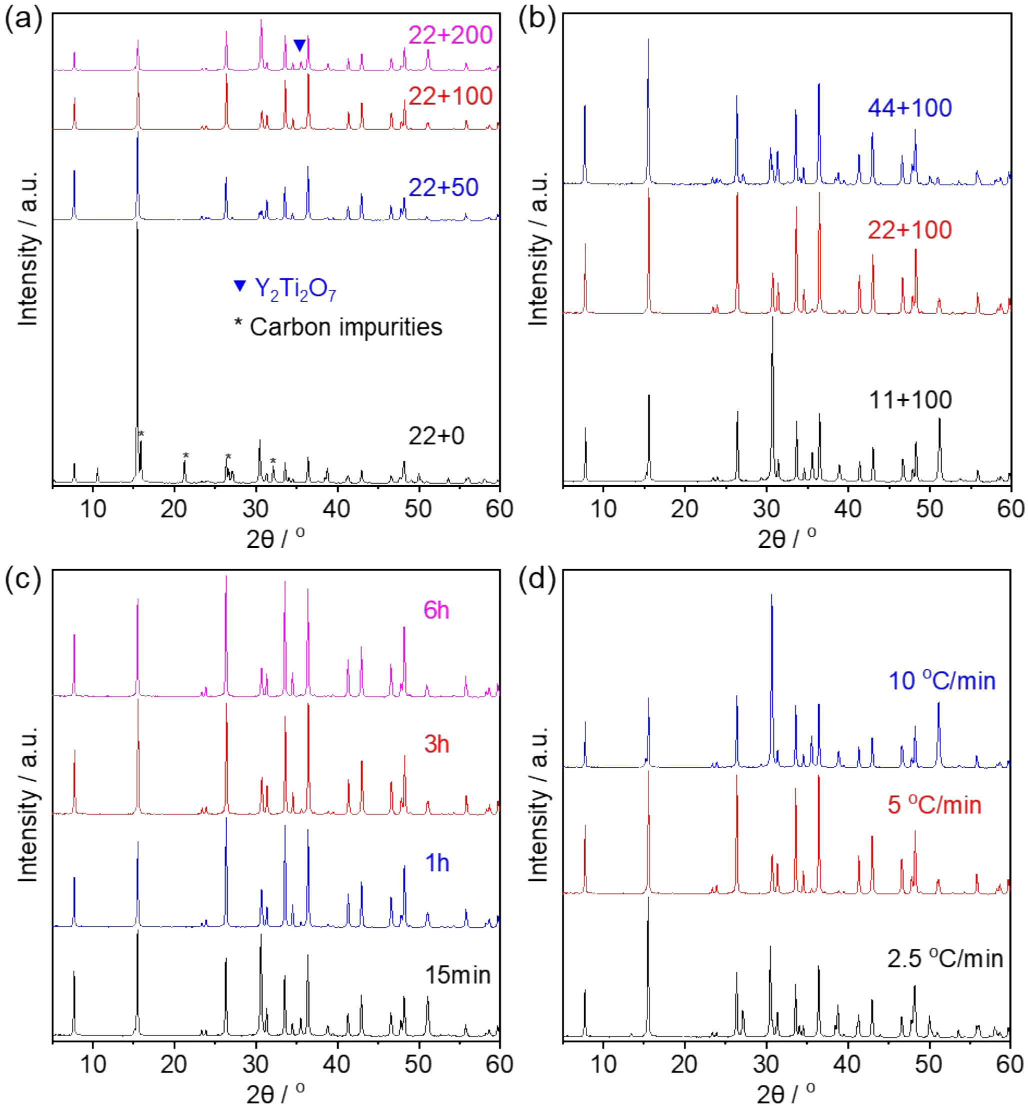

Figure S13. XRD patterns for YTOS-YO specimens synthesized while varying (a) N_2 flow rate, (b) CS_2/N_2 flow rate, (c) reaction duration, and (d) heating rate above 500 °C. The two numbers in panels (a) and (b) indicate the CS_2/N_2 and N_2 flow rates, respectively. Unless noted, the samples were prepared with a CS_2/N_2 flow rate of 22 mL/min and a N_2 flow rate of 100 mL/min and a duration of 3 h at 800 °C with a ramp rate of 10 °C/min to 500 °C followed by 5 °C/min to 800 °C.

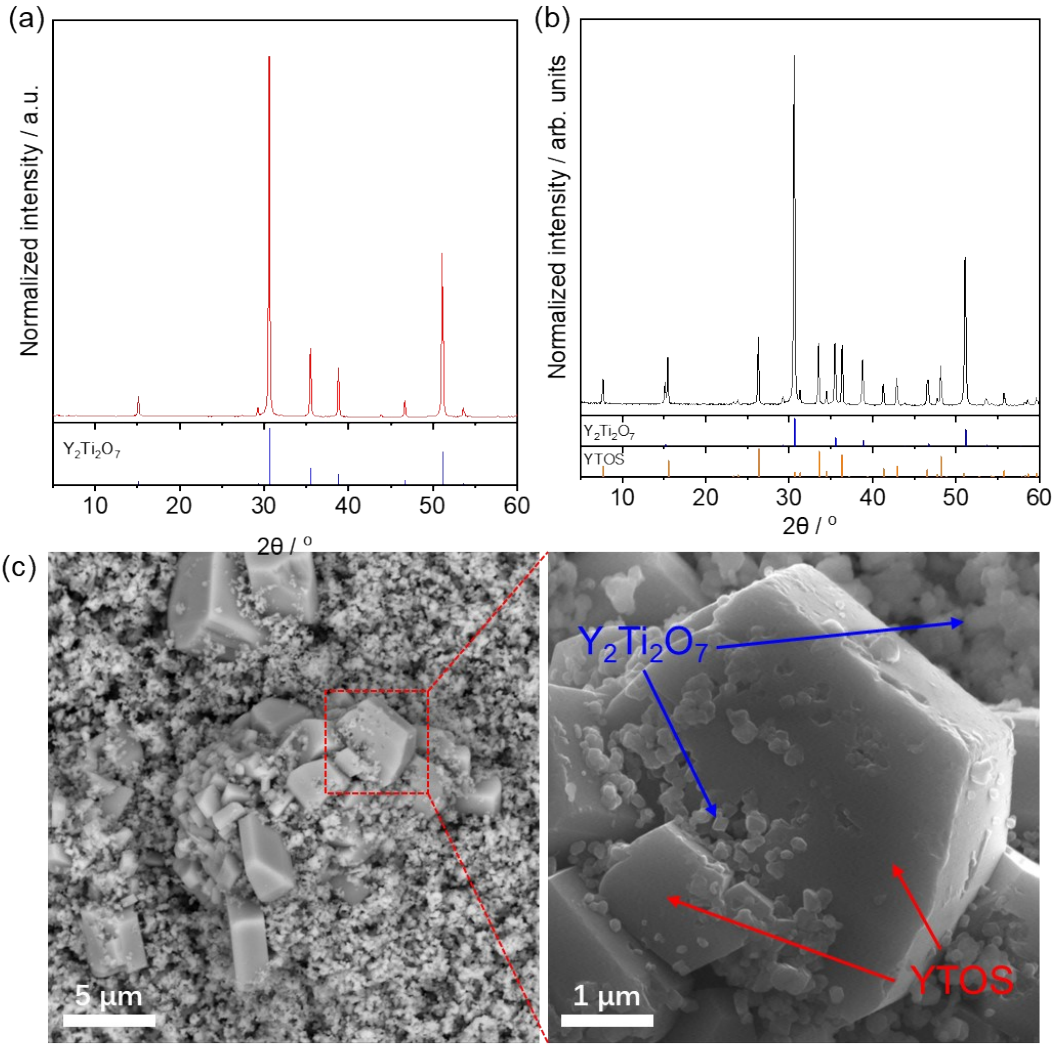

Figure S14. XRD patterns for (a) as-prepared $Y_2Ti_2O_7$ and (b) YTOS-YTO. (c) SEM images of YTOS-YTO.

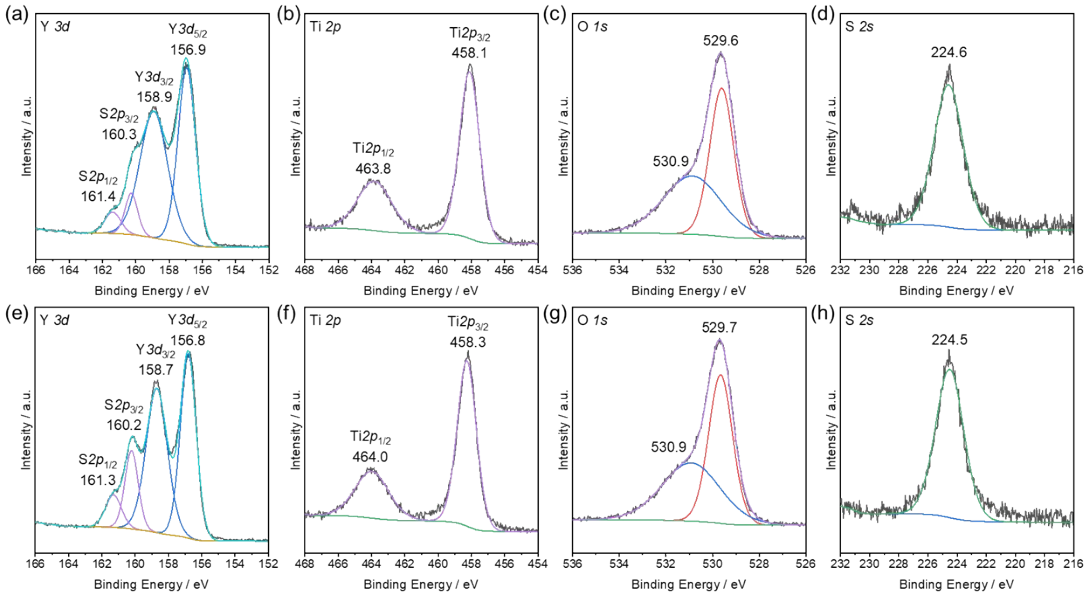

Figure S15. XPS patterns of YTOS-YO with duration of (a-d) 3 h and (e-h) 6 h.

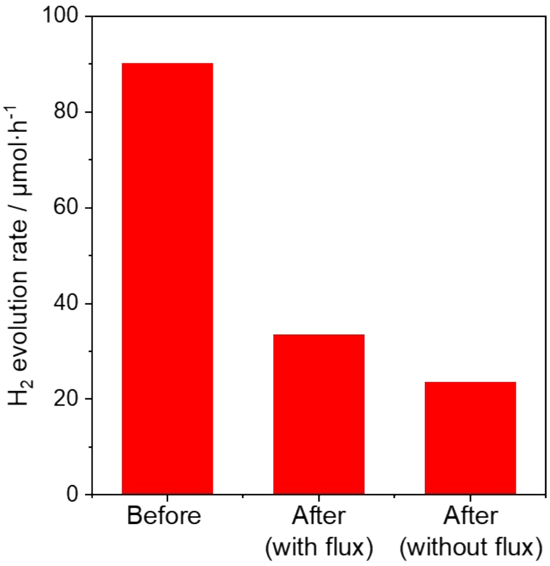

Figure S16. H_2 evolution rates for YTOS-SSR before and after a CS_2 treatment. The treatment was carried out with a CS_2/N_2 flow rate of 22 mL/min and a N_2 flow rate of 100 mL/min used for dilution in the presence/absence of a flux for 3 h. The sample to flux ratio and the loading amount of mixture were 1:5 and 3 g, respectively.

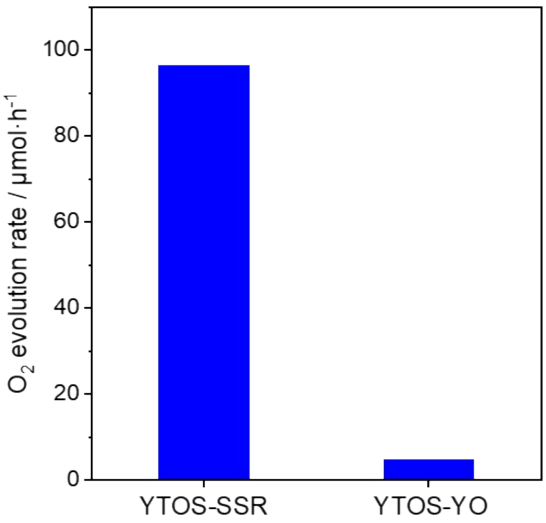

Figure S17. O_2 evolution rates for YTOS-SSR and YTOS-YO specimens prepared with precursor-to-flux ratio of 1:2.

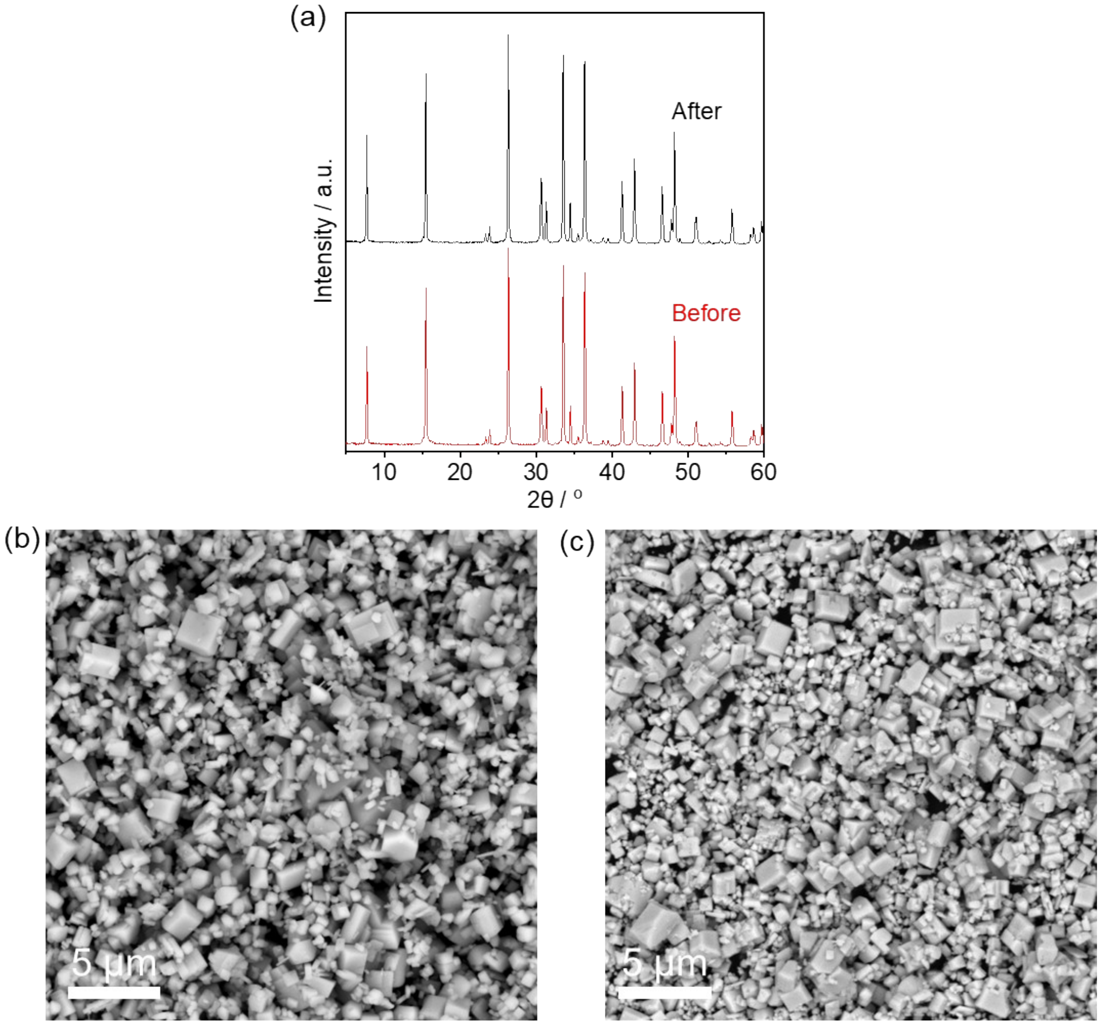

Figure S18. (a) XRD patterns and (b, c) SEM images of YTOS-YO prepared with precursor-to-flux ratio of 1:2 (b) before and (c) after acid treatment.

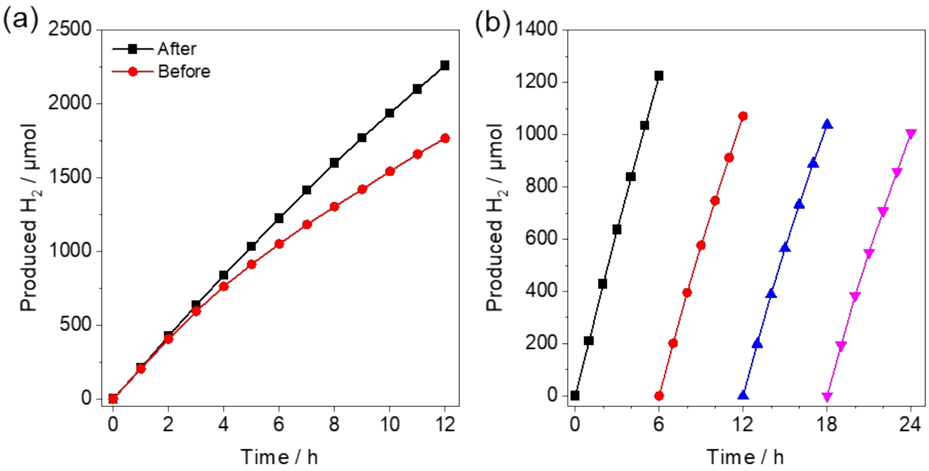

Figure S19. (a) Time-courses of H_2 evolution for YTOS-YO prepared with precursor-to-flux ratio of 1:2 before and after acid treatment. (b) Four cycles of H_2 evolution reaction with evacuation every 6 hours using YTOS-YO sample after acid treatment.

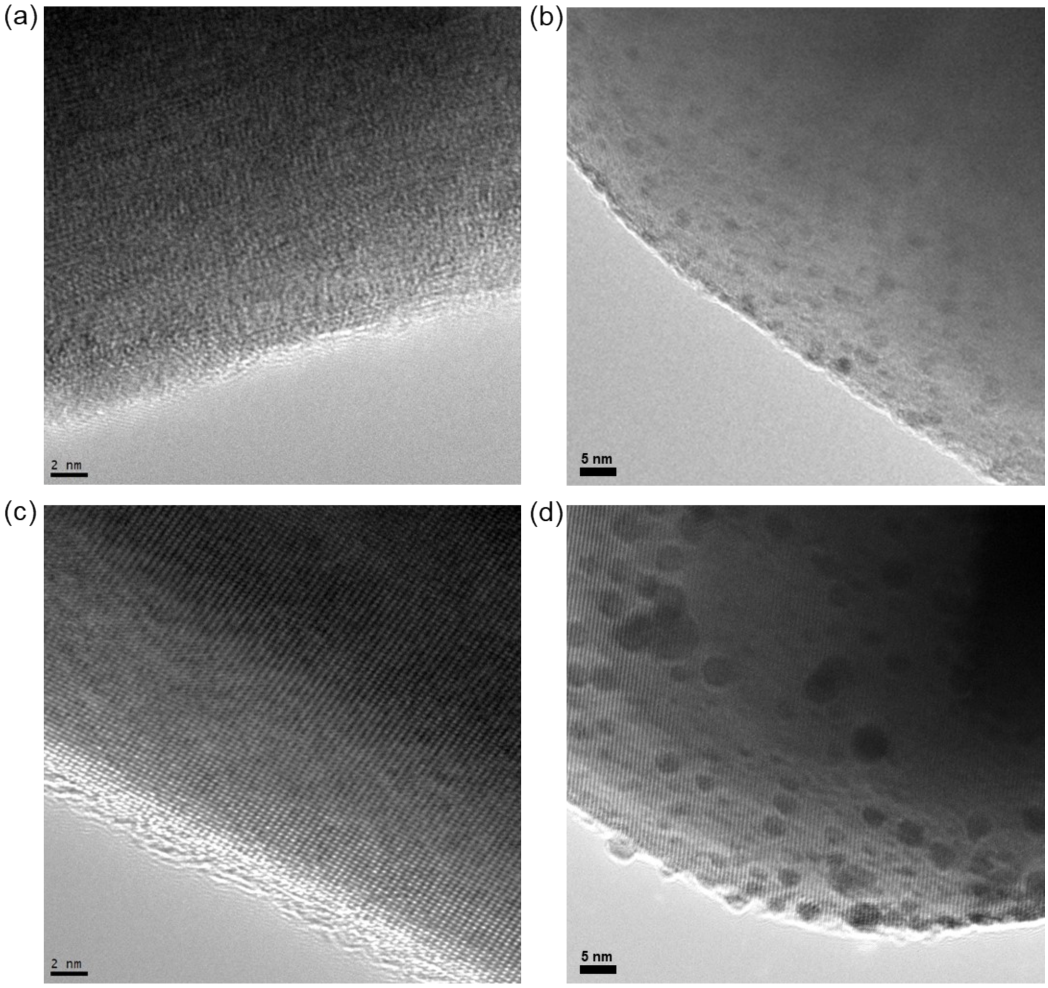

Figure S20. TEM images of the pristine sample (a) without and (b) with Rh cocatalyst deposition, and acid treated sample (c) without and (d) with Rh cocatalyst deposition.

Table S1. H_2 evolution performance of YTOS reported in the literatures and this work.

Entry	Method	Flux	Reactor	H_2 evolution (μmol/h)	Conditions	AQY (420nm)	Ref.
1	H_2S sulfidation	-	Alumina boat	66	2.0 wt% Rh, >420 nm, 20 mM N_2S/Na_2SO_3	-	33
2	Flux-assisted sulfidation with CS_2	$CaCl_2$	Alumina boat	204	1.0 wt% Rh, >420 nm, 20 mM N_2S/Na_2SO_3	3.5%	**This work**
3	SSR	-	Sealed quartz tube	90	1.0 wt% Rh, >420 nm, 20 mM N_2S/Na_2SO_3	1.5%	**This work**
4	SSR with Sc-doped in flux	KI	Sealed quartz tube	445	2.0 wt% Rh, >420 nm, 20 mM N_2S/Na_2SO_3	-	8
5	Flux-assisted	$CaCl_2$	Sealed quartz tube	218	1.0 wt% Rh, >420 nm, 20 mM N_2S/Na_2SO_3	-	**This work**
6	Flux-assisted	$CaCl_2$	Sealed quartz tube	222	1.0 wt% Rh, >420 nm, 20 mM N_2S/Na_2SO_3	-	21
7	Flux-assisted with rapid heating	$CaCl_2$	Sealed quartz tube	387	1.0 wt% Rh, >420 nm, 20 mM N_2S/Na_2SO_3	5.9%	21
8	Flux-assisted	$MgCl_2/CaCl_2$	Sealed quartz tube	1066	2.0 wt% Pt, >420 nm, 20 mM N_2S/Na_2SO_3	10.7%	23