Synergistically Enhanced Photocatalytic Properties of Co₃O₄-G/GO Nanocomposites: Unravelling Their Interactions and Charge Transfer Dynamics Using XAS

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Fig.S1: Tauc plot corresponding to the observed optical of the synthesized samples.



Fig.S2: EDAX mapping of C1.



Fig.S3: EDAX mapping of C2.



Fig.S4: EDAX mapping of C3.



Fig.S5: Photoluminescence spectrum of C1 with deconvoluted peaks.

Experimental conditions for XAS Experiments

The soft X-ray Absorption spectra of the synthesized samples were collected in the powder form at the BL20A1 at the Taiwan Light Source (TLS) in National Synchrotron Radiation Research Centre (NSRRC), Taiwan. The powdered samples were put on copper tape and mounted on the sample holder before loading in the load-lock chamber and transferring to experimental analysis chamber. A vacuum with a base pressure better than 1×10^{-9} Torr was provided in the experimental analysis chamber. Each XAS spectrum edge was aligned to a standard reference sample as per the required energy. The data signal was collected three times in total electron yield (TEY) mode with aligning powder sample normal to the incident X-ray beam and the averaged data was used as raw data. And then the obtained raw data was processed following the steps mentioned below:

Step1: The obtained raw data of the sample was calibrated for the threshold energy with the standard reference sample.

Step 2: Then, a linear background subtraction was performed using the pre-edge region.

Step 3: Then, absorption intensity of the spectra was adjusted by normalising it at the post-edge absorption intensity.



A schematic for the XAS experimental setup is shown below in fig.S6.

Fig.S6 Schematic of the XAS performed at BL20A1, NSRRC.

Experimental conditions for in-situ XAS Experiments

The *in-situ* XAS under light (using AM 1.5 solar simulator lamp) was collected at the BL20A1 at the Taiwan Light Source (TLS) in National Synchrotron Radiation Research Center (NSRRC), Taiwan. The powdered samples were put on copper tape and mounted on the sample holder before loading in the load-lock chamber and transferring to experimental analysis chamber. A vacuum with a base pressure better than 1×10^{-9} Torr was provided in the analysis chamber. The signal was collected minimum two times in total electron yield (TEY) mode with aligning powder sample normal to the incident x-ray beam. And the averaged data was considered as the obtained spectrum. An AM 1.5 Solar simulator was positioned with its focus on the sample through an FSQ-KG5 optical filter to allow only desired energy of light to pass through it. The data was detected in two conditions of the solar simulator (Lamp ON state-lamp illuminating state, and Lamp OFF state-dark state). The data was collected in a sequential manner in both the lamp ON and lamp OFF states, and this process was iterated three times to mitigate potential sources of error in the measurements. This approach was implemented to

ensure robustness and enhance the reliability of the collected data. And then the obtained raw data in lamp On and Off state was processed using the following steps:

Step1: The obtained data of the sample was calibrated for the threshold energy with the standard reference sample.

Step 2: Then, a linear background subtraction was performed using the pre-edge region.

Step 3: Then, absorption intensity of the spectra was adjusted by normalising it at the postedge absorption intensity.

A schematic for the *in-situ* XAS experimental setup is shown below in fig.S7. The background signals (without any sample) were measured in the light illumination and dark conditions for the O *K*-edge and Co $L_{3,2}$ -edge but no edge jump or absorption spectra was observed for any edge.



Fig.S7. Schematic of the *in-situ* XAS performed under Lamp off and on conditions using solar simulator.