Supplementary Material (ESI) for *Energy & Environmental Science*

Titanium-Embedded Layered Double Hydroxides as Highly Efficient Water Oxidation Photocatalysts under Visible Light

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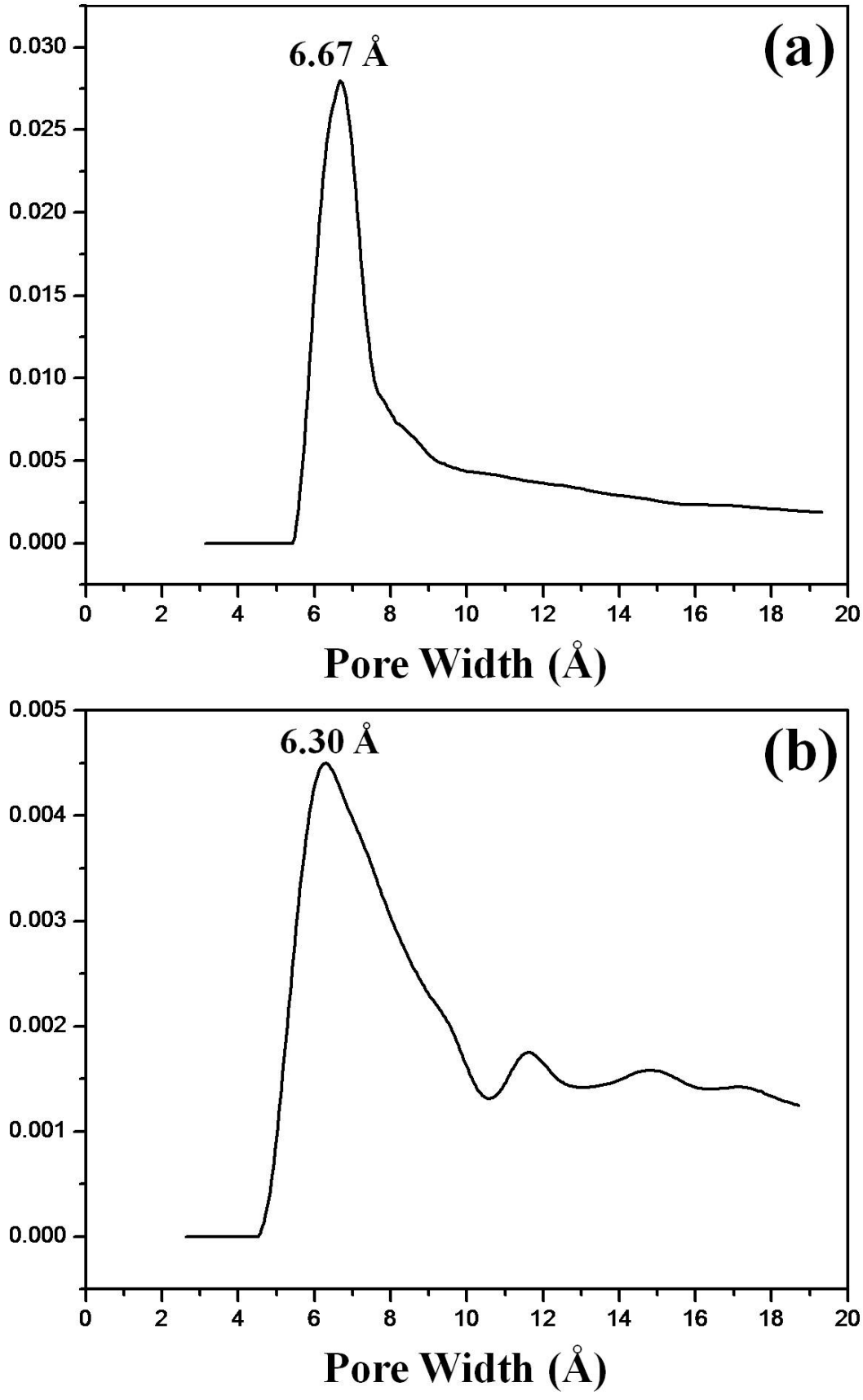
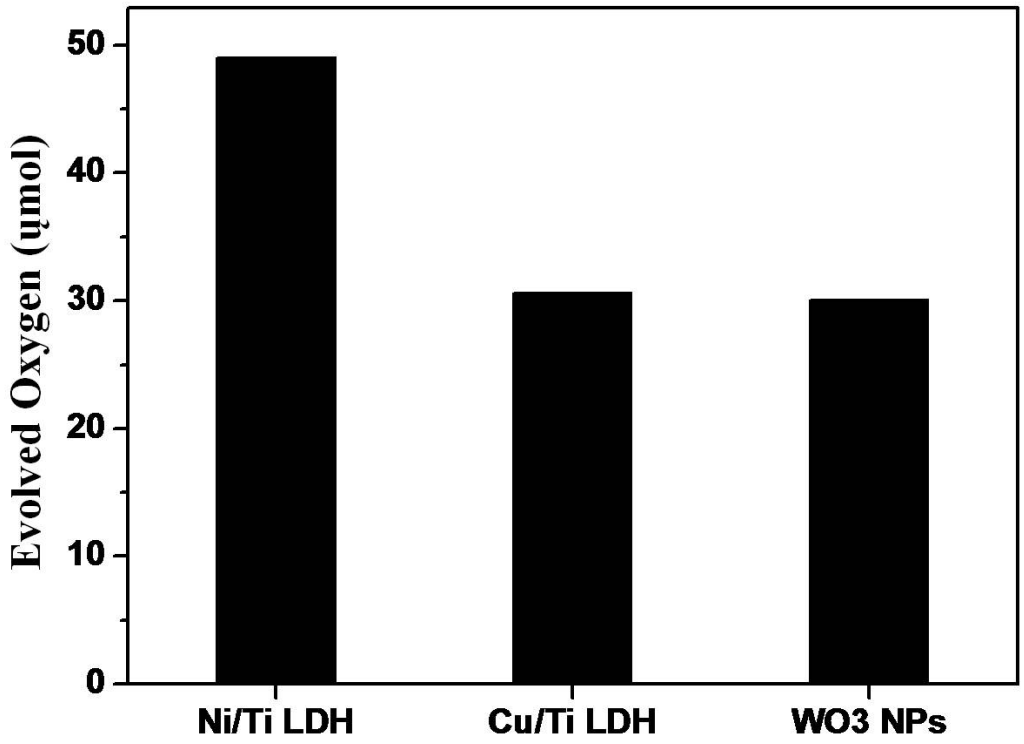
Supplementary Information

Fig. S1 Pore distributions of (a) (Ni/Ti)LDH and (b) (Cu/Ti)LDH



The pore distribution of the two materials is calculated by HK method based on low temperature N_2 sorption hysteresis (Fig. 5). The peak is located at 6.67 Å for (Ni/Ti)LDH and 6.30 Å for (Cu/Ti) LDH. These values are similar to the values by calculated from (003) peak of powder x-ray diffraction pattern (Fig. 2).

Fig. S2 Amounts of evolved oxygen from the aqueous suspension on the three types of photocatalysts

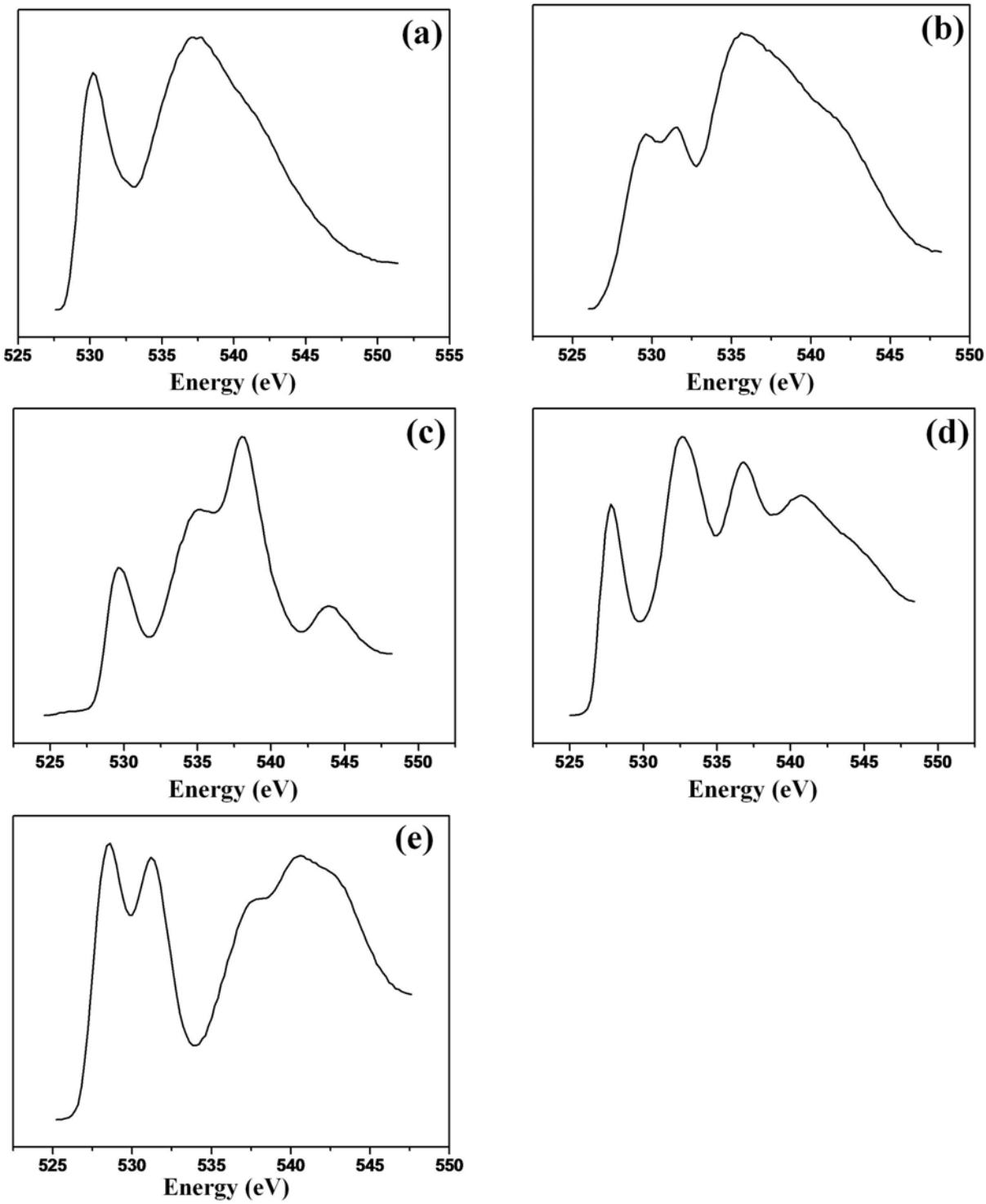


The photocatalytic analysis of WO_3 nanopowders was conducted in same conditions for two LDHS. The 125 mL Pyrex flask contained the 100 mL of the aqueous suspension. The headspace was connected to an inverted burette filled with water at atmospheric pressure to make a closed system. The photocatalyst was degassed overnight in a Schlenk line under vacuum and refilled with Ar before being transferred to the reactor. The aqueous suspension consisted of 200 mg of WO_3 nanopowders and 1 mmol of $AgNO_3$ (99%) as sacrificial agent. The suspensions were purged with Ar for 1 h to remove remaining atmospheric air in the reactor. Then, the reactor was irradiated with a 300 W xenon lamp. Two types of filter were used for visible light irradiation (400 nm < λ < 700 nm). 0.5 mL of gas was periodically captured from the headspace of the reactor and this gas was then directly injected into a gas chromatograph (HP 7890) to confirm the production of oxygen by photocatalysis. The gas

chromatograph was operated under isothermal conditions (60 ºC) using a packed column (molecular sieve 5A, 3 mm diameter, 3 m length) equipped with a thermal conductivity detector.

The WO_3 nanopowders generated 30 ų moles of oxygen under visible light. This value is 60 % of the value of generated oxygen by the (Ni/Ti)LDH and similar to the value of generated oxygen by the (Cu/Ti)LDH.

Fig. S3 The oxygen K-edged NEXAFS data of (a) (Ni/Ti)LDH, (b) (Cu/Ti)LDH, (c) NiO, (d) CuO, (e) TiO_2



The oxygen K-edge Near Edge X-ray Absorption Fine Structure (NEXAFS) data were collected by Pohang accelerator in Pohang, Republic of Korea. There are clear differences between the data of LDHs and the data of the pure oxides. Therefore, we find that the bonding nature of oxygen in LDHs is not same with the pure oxides. It assists that the synthesized LDHs consist of the M^{II}-O-Ti^{4+} bonding.