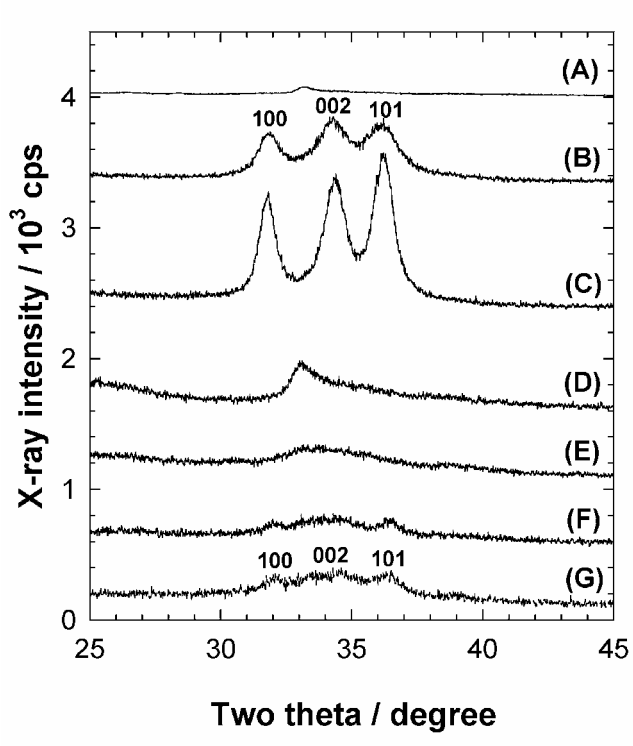
Supplementary Material (ESI) for Chemical Communications

Supplementary Information

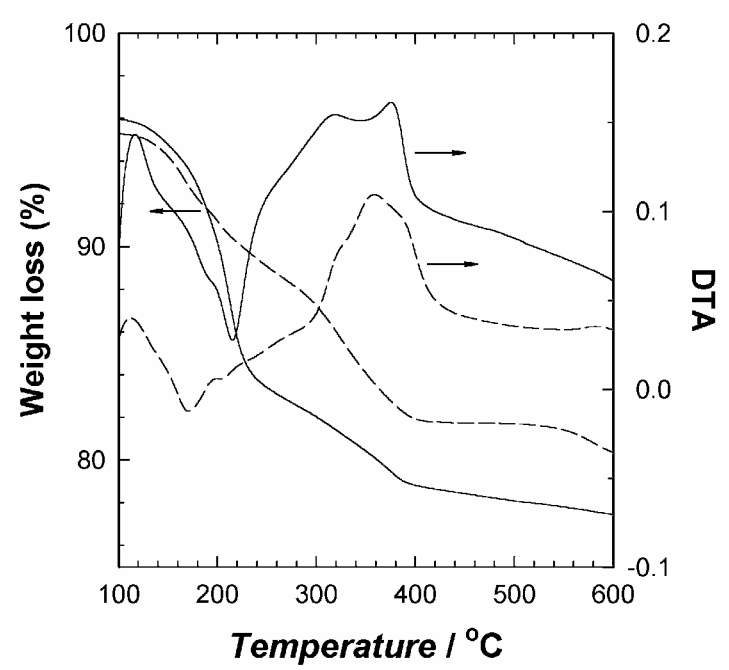
: For the manuscript entitled with "Layered titanate–zinc oxide nanohybrids with mesoporosity" by *Tae Woo Kim, Su Gil Hur, Seong-Ju Hwang*, and Jin-Ho Choy**

1. XRD patterns in high angle region



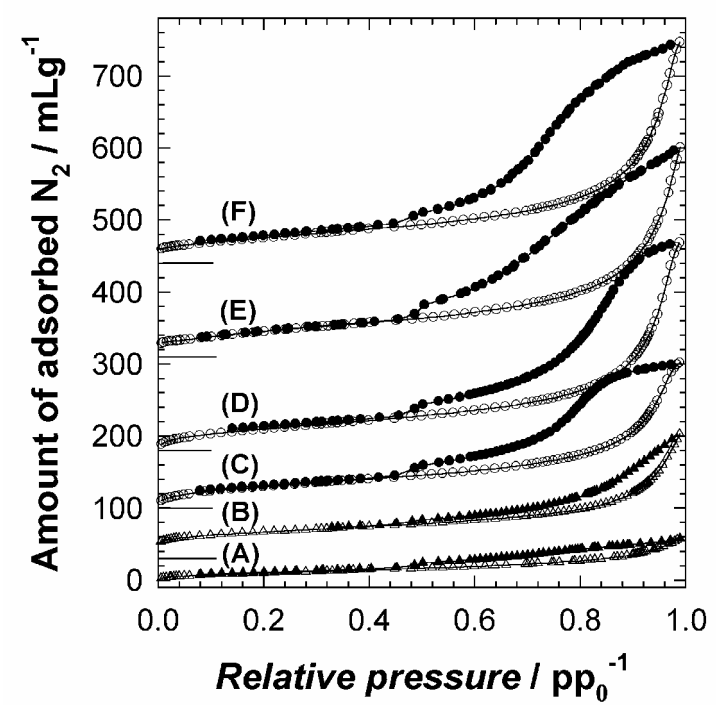
: The above figure presents power XRD patterns in high angle region for (A) ZnO–$Ti_{1.83}O_4$-**R** and its derivative calcined at (B) 200 and (C) 400 °C, (D) ZnO–$Ti_{1.83}O_4$-**H** and its derivatives calcined at (E) 200, (F) 300, and (G) 400 °C. Each pattern is shift along the *y*-axis for the clarity. After the heat-treatment at 200–400 °C, the ZnO–$Ti_{1.83}O_4$-**R** shows well-developed peaks corresponding to ZnO at high angle region of 30–40°, clarifying the formation of bulk ZnO particles. On the contrary, the calcined derivative at 200 °C of the ZnO–$Ti_{1.83}O_4$-**H** does not show these ZnO peaks whereas they are observable with very weak and diffusive features after the heat-treatment at 400 °C. The weak intensity of these reflections provides clear evidence on the nanocrystalline nature of ZnO particles formed on the calcined derivative of the hydrothermally prepared sample, as suggested from the UV–vis analysis.

2. TG–DTA results



: The thermal behavior of the ZnO–$Ti_{1.83}O_4$ nanohybrids was probed by performing TG–DTA analysis under ambient atmosphere. As shown in the above figure, the ZnO–$Ti_{1.83}O_4$-**R** (solid lines) and ZnO–$Ti_{1.83}O_4$-**H** (dashed lines) nanohybrids exhibit considerable weight loss below 400 °C. A weight decrease in the temperature range of 100–250 °C is attributable to the dehydration and the dehydroxylation of the nanohybrids. The decomposition of residual organic group in the interlayer zinc species is responsible for a weight loss in higher temperature region. No marked weight loss occurs beyond 400 °C.

3. N_2 adsorption–desorption isotherm results



: The above figure presents nitrogen isotherms of (A) ZnO–$Ti_{1.83}O_4$-**R** and (B) its derivative calcined at 200 °C, (C) ZnO–$Ti_{1.83}O_4$-**H** and its derivatives calcined at (D) 200, (E) 300, and (F) 400 °C. Each isotherm is shift along the *y*-axis for the clarity. The curve shape of all of the present isotherms can be classified as the BDDT type I and IV, suggesting the presence of mesoporous adsorbents. In addition, the hysteresis loop resembles H4 in the IUPAC classification, indicative of the open slit-shaped capillaries with very wide bodies and narrow short necks. A weak adsorption of N_2 molecules in a low relative pressure (p/p_0) region and a distinct hysteresis in the region of $p/p_0 > 0.5$ underline that most of porosity in the present nanohybrids originates from mesopores in the stacked structure of nanohybrid crystallites. It is worthwhile to note here that, in spite of the collapse of the heterostructure, the calcined derivative of ZnO–$Ti_{1.83}O_4$-**H** at 400 °C still shows a marked hysteresis and an expanded surface area with mesoporosity, underlining the high stability of pore-structure in the hydrothermally prepared nanohybrids. Therefore, these mesoporous materials are expected to be useful as efficient catalysts or absorbents.