Facile microwave-assisted synthesis and controllable architecture of three-dimensional nickel titanate

Thuy-Duong Nguyen-Phan,[a] Chinh Nguyen Huy[a] Chang-Koo Kim[b] and Eun Woo Shin*[a]

[a] School of Chemical Engineering, University of Ulsan, Daehakro 93, Nam-gu, Ulsan 680-749, South Korea.

[b] Department of Chemical Engineering and Energy Systems Research, Ajou University, 206 Worldcup-ro, Yeongtong-gu, Suwon 443-749, South Korea.

Corresponding Author

*Email address: ewshin@mail.ulsan.ac.kr. Fax: +82 52 259 1689; Tel.: +82 52 259 2253

Electronic Supplementary Information. Experimental detail of solvolthermal synthesis, methylene blue decolorization and nitrobenzene degradation under visible light illumination. Figures S1-S5 include Tauc's plot for band gap determination, nitrobenzene photodegradation, methylene blue photodecolorization, N_2 sorption isotherm and crystal structure of $NiTiO_3$.

1. Solvothermal synthesis of nickel titanate

A mixture containing 0.01 mol of $Ti(O\text{-}C_4H_9)_4$, 0.01 mol of $Ni(NO_3)_2.6H_2O$ and 50 mL of $C_2H_6O_2$ (99.9%) was vigorously stirred at room temperature for 8 h. Subsequently, the solution was transferred into a Teflon-lined stainless steel autoclave, and kept for 12 h at 200 °C. A light green precipitate was subsequently collected by centrifugation and washed several times with ethanol. The product was dried at 80 °C overnight and then calcined in air at 600 °C for 4 h. The final yellow nickel titanate was denoted as "**NTN-N-EG-s**".

2. Photodegradation of nitrobenzene

Photocatalytic activity was evaluated through the degradation of nitrobenzene (NB). 50 mg of NTN material was immersed in 100 mL of aqueous NB solution (C_o = 10 ppm). The photoreactor was placed in a dark chamber equipped with a cooling fan under constant stirring. After 90 min of dark adsorption to reach equilibrium, four surrounding 3-W blue LED lamps (CR Lighting Technology Co., Ltd, China) with a lumin flux of 210 Lm and a wavelength range of 450-500 nm were then switched on to illuminate the photoreactor. The concentration of NB upon light exposure was monitored by observing changes in the absorbance at λ_{max} = 264 nm as a function of irradiation time using a UV-Vis absorbance microplate spectrophotometer (Spectra Max® Plus 384).

3. Photocatalytic decolorization of methylene blue

The photocatalytic decolorization of methylene blue was determined by using a UV-Vis absorbance microplate spectrophotometer (Spectra Max® Plus 384). 20 mg of catalyst were immersed into 50 mL of an aqueous methylene blue (C_o = 2.5x10^{-5} M) solution under constant stirring. After 1 hour-reaching the adsorption equilibrium in dark chamber, the solution containing catalyst was irradiated by four 20 W tubular-like compact fluorescent lamps (λ_{max} = 545 nm). The changes in dye concentration during photodecolorization were characterized by the absorbance at λ_{max} = 664 nm as a function of irradiated time.

4. Supporting figures

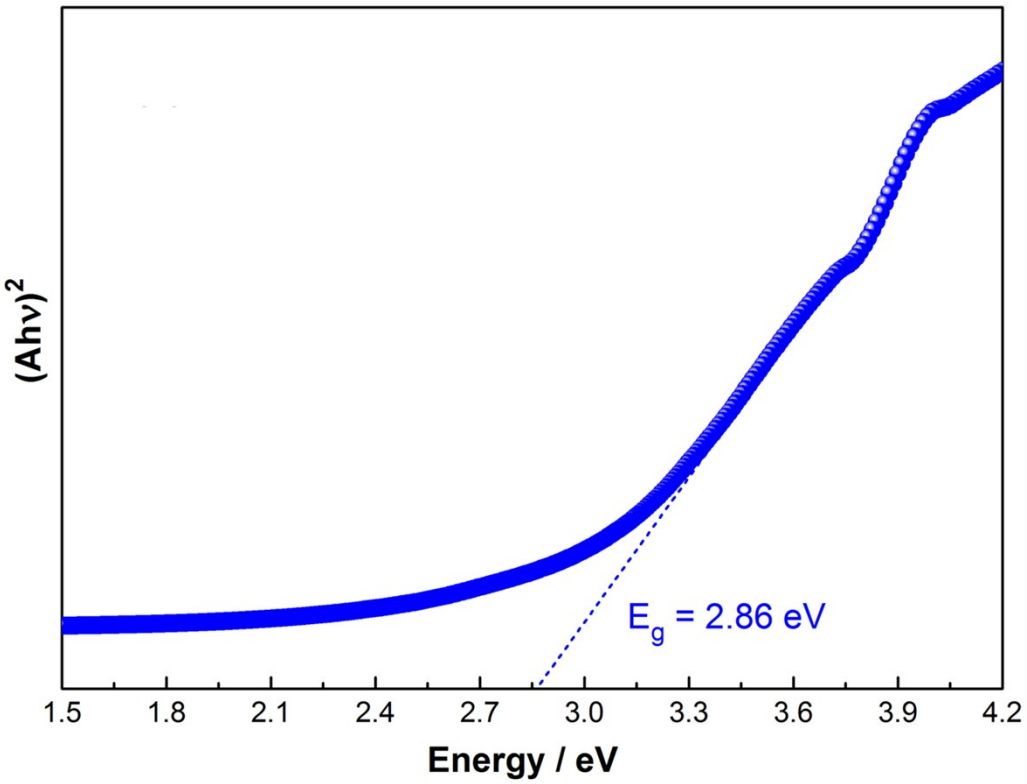

Figure S1. Tauc's plot for band gap determination of NTN-N-EG derived from UV-Vis-DRS spectrum.

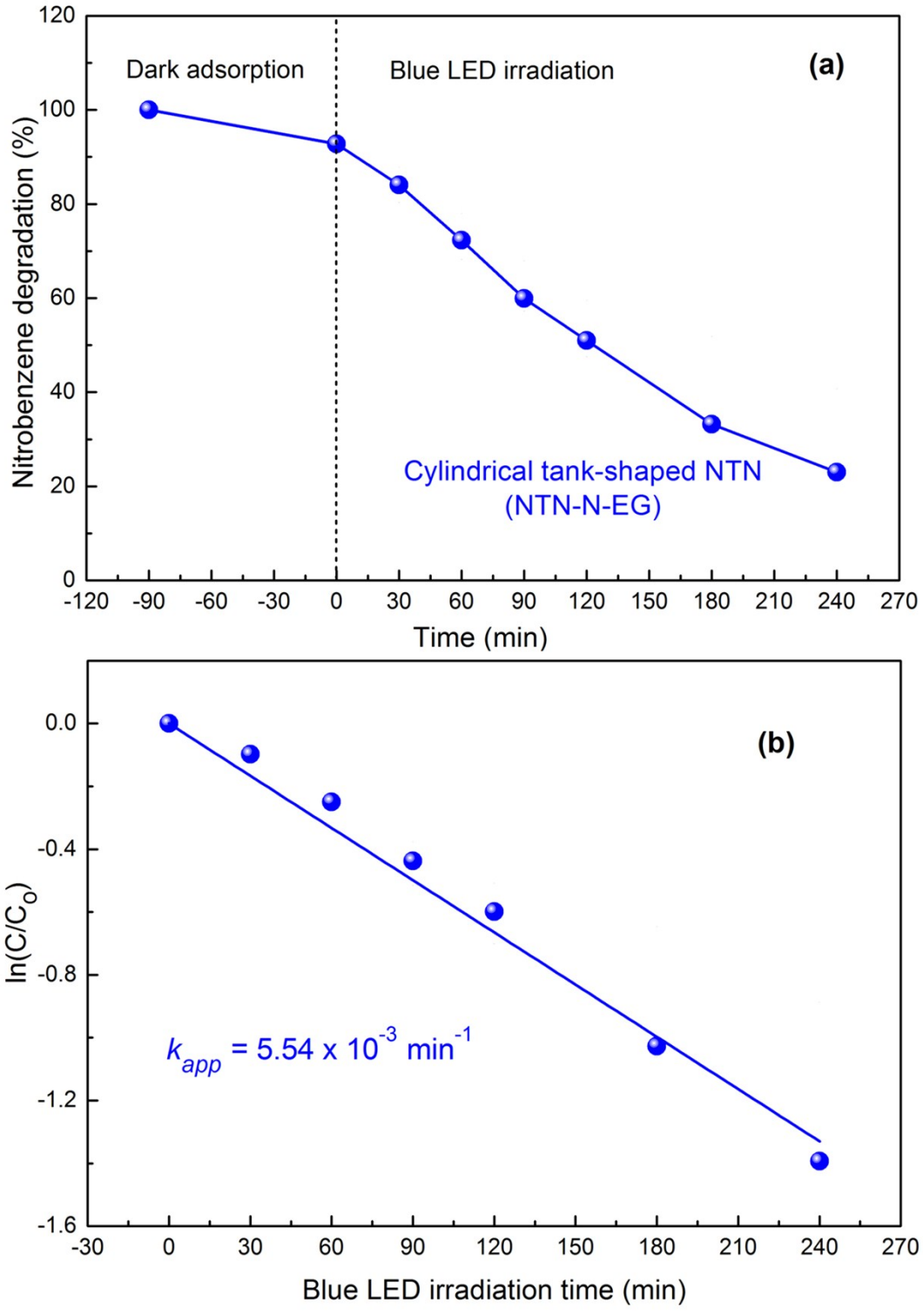

Figure S2. (a) Photocatalytic degradation of nitrobenzene and (b) pseudo-first-order kinetics of nitrobenzene photodegradation cylindrical tank-like nickel titanate upon blue LED irradiation.

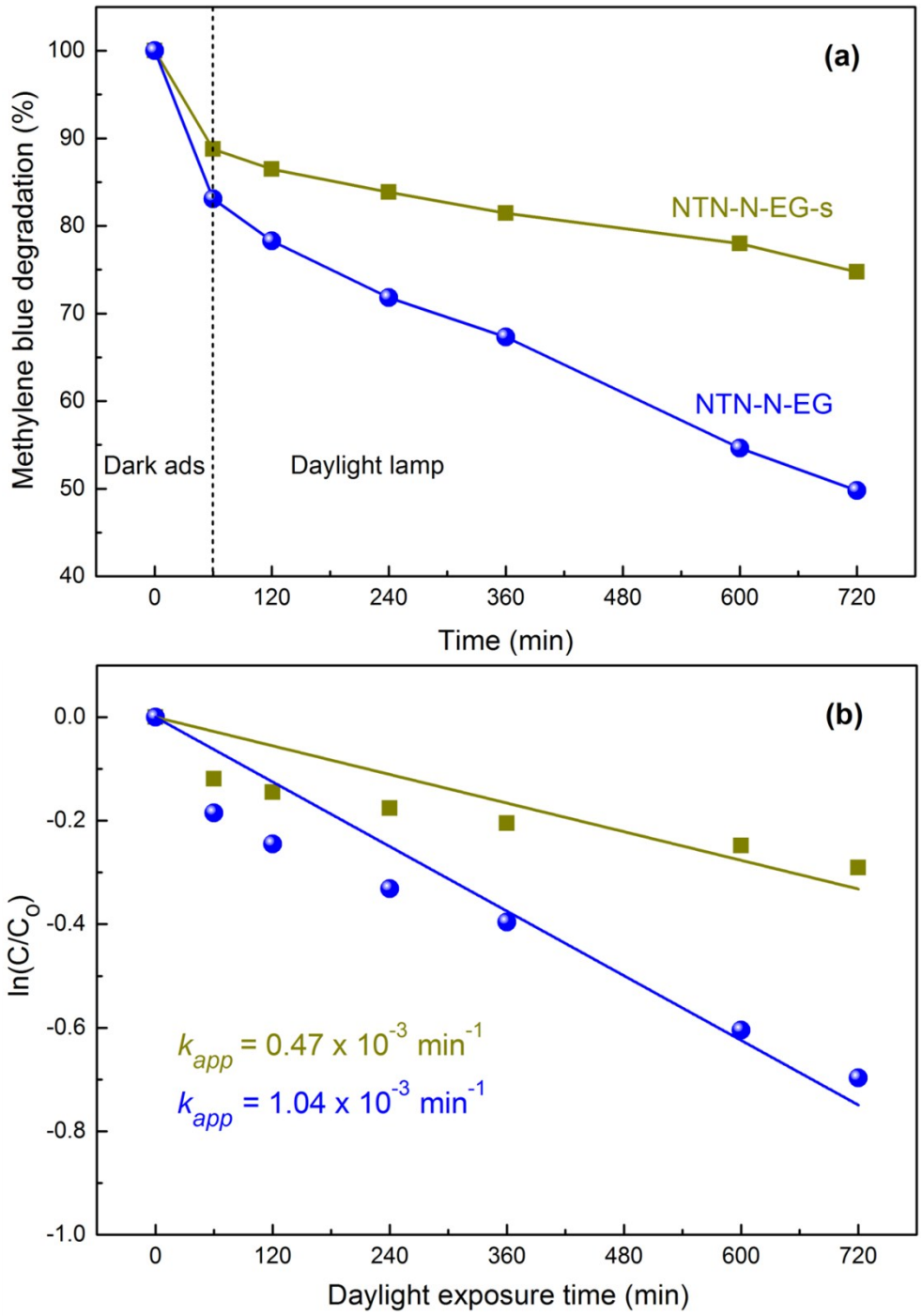

Figure S3. (a) Photocatalytic decolorization of methylene blue (10 ppm); (b) pseudo-first-order kinetics over NTN-N-EG prepared by solvothermal and microwave synthesis, respectively, under the illumination of tubular-like compact fluorescent lamp. It can be seen that 3D architecture is beneficial to the photoactivity in comparison with the irregular geometry.

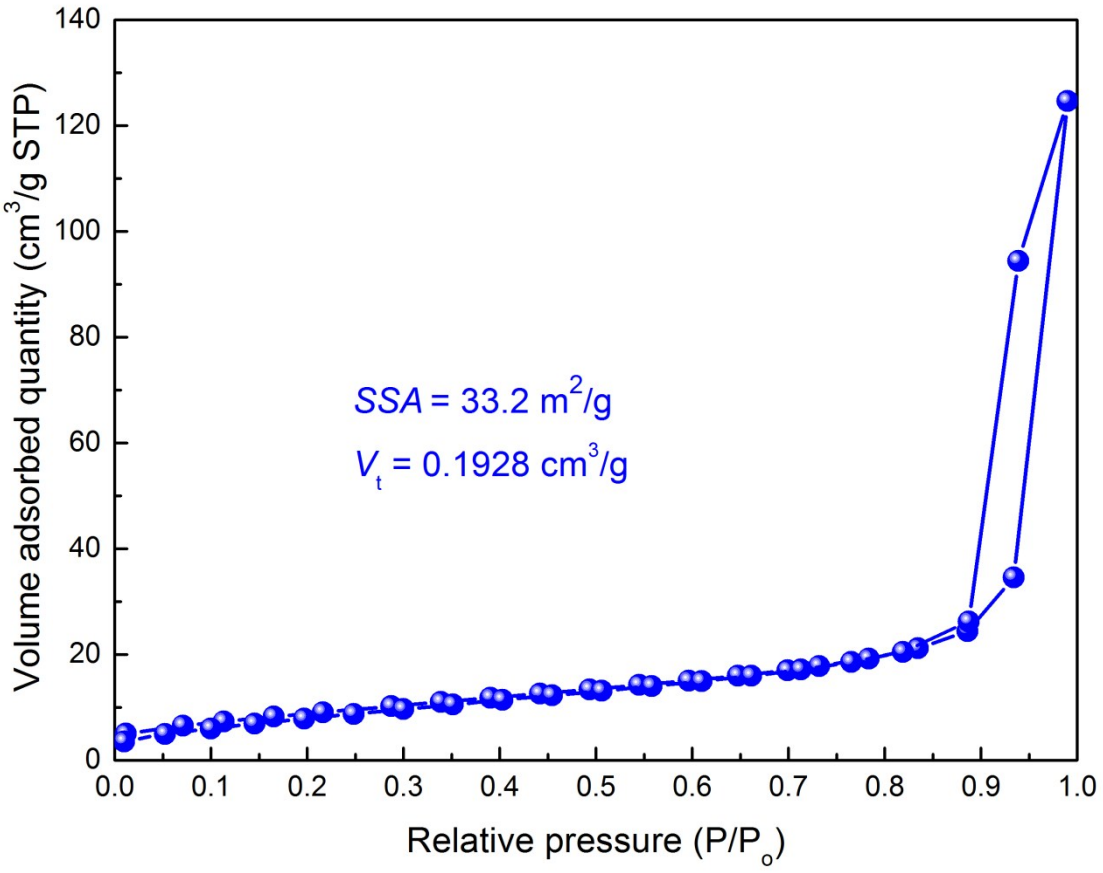

Figure S4. N_2 adsorption-desorption isotherms of NTN-EG.

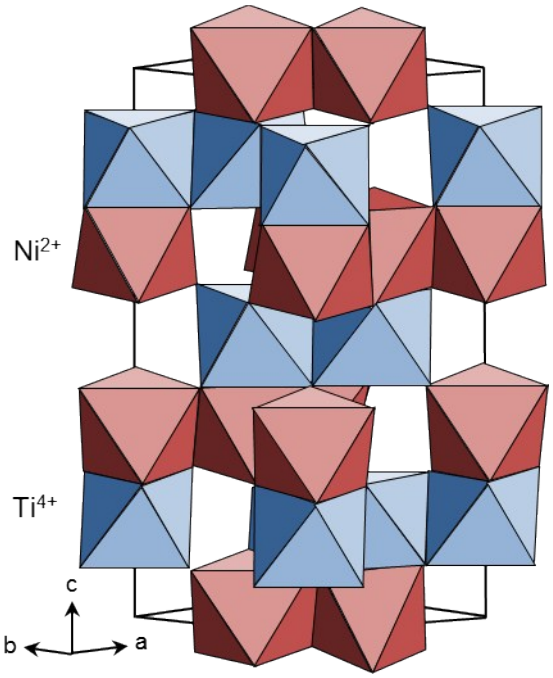

Figure S5. Crystal structure of $NiTiO_3$.