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Role of Calcination Treatment on the Structural Properties and Photocatalytic Activity of Nanorice N-Doped TiO_2 Catalyst

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Abstract : The purposes of this research were to synthesize titanium dioxide photocatalyst doped with nitrogen (N-doped TiO_2) by hydrothermal method and to test the photocatalytic degradation of paraquat under UV and visible light illumination. The effect of calcination treatment temperature on their physical and chemical properties and photocatalytic efficiencies were also investigated. The characterizations of calcined N-doped TiO_2 photocatalysts such as specific surface area, textural properties, bandgap energy, surface morphology, crystallinity, phase structure, elements and state of charges were investigated by Brunauer, Emmett, Teller (BET) and Barrett, Joyner, Halenda (BJH) equations, UV-Visible diffuse reflectance spectroscopy (UV-Vis-DRS) by using the Kubelka-Munk theory, Wide-angle X-ray scattering (WAXS), Focussed ion beam scanning electron microscopy (FIB-SEM), X-ray photoelectron spectroscopy (XPS) and X-ray absorption spectroscopy (XAS), respectively. The results showed that the effect of calcination temperature was significant on surface morphology, crystallinity, specific surface area, pore size diameter, bandgap energy and nitrogen content level, but insignificant on phase structure and oxidation state of titanium (Ti) atom. The N-doped TiO_2 samples illustrated only anatase crystalline phase due to nitrogen dopant in TiO_2 restrained the phase transformation from anatase to rutile. The samples presented the nanorice-like morphology. The expansion on the particle was found at 650 and 700°C of calcination temperature, resulting in increased pore size diameter. The bandgap energy was determined by Kubelka-Munk theory to be in the range 3.07-3.18 eV, which appeared slightly lower than anatase standard (3.20 eV), resulting in the nitrogen dopant could modify the optical absorption edge of TiO_2 from UV to visible light region. The nitrogen content was observed at 100, 300 and 400°C only. Also, the nitrogen element disappeared at 500°C onwards. The nitrogen (N) atom can be incorporated in TiO_2 structure with the interstitial site. The uncalcined (100°C) sample displayed the highest percent paraquat degradation under UV and visible light irradiation due to this sample revealed both the highest specific surface area and nitrogen content level. Moreover, percent paraquat removal significantly decreased with increasing calcination treatment temperature. The nitrogen content level in TiO_2 accelerated the rate of reaction with combining the effect of the specific surface area that generated the electrons and holes during illuminated with light. Therefore, the specific surface area and nitrogen content level demonstrated the important roles in the photocatalytic activity of paraquat under UV and visible light illumination.

Keywords : restraining phase transformation, interstitial site, chemical charge state, photocatalysis, paraquat degradation
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