University of Victoria | Graduate Studies

Notice of the Final Oral Examination
for the Degree of Doctor of Philosophy

of

MOHAMMAD ALI PELASCHI

MSc (Sharif University of Technology, 2011)
BSc (Sharif University of Technology, 2008)

"Synthesis and Characterization of Nitrogen-Doped Titanium Oxide Nanoparticles for Visible-Light Photocatalytic Wastewater Treatment"

Department of Mechanical Engineering

Friday, September 14, 2018
12:00 P.M.
Elliott Building
Room 305

Supervisory Committee:
Dr. Martin Jun, Department of Mechanical Engineering, University of Victoria (Co-Supervisor)
Dr. Frank van Veggel, Department of Chemistry (Co-Supervisor)
Dr. Rustom Bhiladvala, Department of Mechanical Engineering, UVic (Member)
Dr. Peter Wan, Department of Chemistry, UVic (Outside Member)

External Examiner:
Dr. Howon Lee, Department of Mechanical and Aerospace Engineering, Rutgers University

Chair of Oral Examination:
Dr. Byoung-Chul Choi, Department of Physics and Astronomy, UVic

Dr. David Capson, Dean, Faculty of Graduate Studies

Abstract

TiO_2 nanoparticles are one of the most suitable materials for photocatalysis, specifically for water and air treatment and removal of a wide variety of organic pollutants such as dyes, aromatic compounds, and chlorinated aromatic compounds. Methods of synthesis of TiO_2 are generally categorized in two main classes of wet chemical, and dry methods. Wet chemical methods generally provide a better control over size, size distribution, and shape; all of which significantly affect photocatalytic performance of the produced nanoparticles. Despite its advantages over other semiconductor photocatalysts, wide band-gap of titania restrains its photocatalytic activity to only UV light, which only makes up to 5% of the light reaching surface of the earth. To induce visible-light activity, titania has been doped by different dopants, including transition metal-dopants such as Fe, and Co and non-metal dopants such as N, and C. Nitrogen has been shown to be a better dopant, providing a suitably placed energy state within the band-gap of TiO_2, and not suffering from issues related to transition-metal dopants such as low thermal and physical stability and high electron-hole recombination rates. To dope titania with nitrogen, one could add the nitrogen source together with other precursors during synthesis, referred to as wet chemical doping methods, or anneal the synthesized titania nanoparticles under a flow of ammonia at high temperatures, referred to as dry doping methods. While different doping methods have been studied individually, the author maintains that there has been an absence of research comparing the effectiveness of these methods, on photocatalytic performance of *N*-doped TiO_2 within a consistent experiment. In this research TiO_2 nanoparticles were synthesized by a facile, inexpensive sol-gel method, and doping was done by wet chemical methods, dry methods, and a combination of both these methods. Visible-light photocatalytic activity of these nanoparticles was evaluated by their efficiency in degradation of methyl orange. The results show wet doping methods increase the efficiency of titania nanoparticles more than dry doping, or combination of both. Further investigation showed that the main reason for higher activity of wet chemically doped nanoparticles is due to their higher available surface area of 131.7 $m^2.g^{-1}$. After normalizing the available surface area, measured by the BET method, it was shown that a combination of wet chemical doping, and dry doping at 600°C result in the most active nanoparticles, but high temperature dry doping severely decreases the surface area, lowering the overall efficiency of the product. Additionally, *N*-doped TiO_2 nanoparticles were synthesized using a simple hydrothermal method, in which the nitrogen source was used not

only to dope, but also to control shape, size, size distribution, and morphology of the titania nanoparticles, and to induce aqueous colloidal stability. It was shown that addition of triethylamine during the synthesis, results in ultra-small, colloidally stable, cubic TiO_2 nanoparticles, while using triethanolamine results in formation of TiO_2 pallets, assembled into spherical, rose-like structures. The synthesized nanoparticles show impressive efficiency in visible-light removal of phenol, 4-chlorophenol, and pentachlorophenol, achieving 100% degradation of a 100-ppm phenol solution in 90 min, more than 98% degradation of a 20-ppm 4-chlorophenol solution in 90 min, and 97% degradation of a 10-ppm pentachlorophenol in 180 min with 500 ppm loading of the catalyst in all cases. Moreover, synthesized nanoparticles showed no sign of deactivation after 5 consecutive runs, removing 4-chlorophenol, showing their reusability.