THESIS INFORMATION

Title: **STUDY ON PREPARATION OF TiO_2/HYDROXYAPATITE MATERIAL AND ITS APPLICATIONAS PHOTOCATALYST**

Major: Chemical engineering of inorganic compounds

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PhD Student: Nguyen Thi Truc Linh

Advisors: Assoc. Prof. Dr. Phan Dinh Tuan

Dr. Nguyen Van Dung

University: University of Technology, Vietnam National University – Ho Chi Minh City

Abstract:

The thesis deals with the main objectives: (1) Preparation of photo-catalytic TiO_2/Hydroxyapatite (TiO_2/HAp) materials from Vietnamese Ilmenite ore and from commercial TiO_2 Millenium; (2) Determination of their structural characteristics and photo-catalytic activities, and then explaining about the photo-catalytic increase of TiO_2/HAp; (3) Preparation of the photo-catalytic TiO_2/HAp coatings from the TiO_2/HAp powders by using inorganic binders.

The TiO_2 and HAp materials were separately prepared: TiO_2 was prepared from Ilmenite ore by sulphate method; meanwhile, HAp was synthesized by both precipitation and hydrothermal methods. Then, photo-catalytic TiO_2/HAp materials were prepared by the methods similar to those of HAp materials.

The characteristics of the materials were determined by X-ray Photoemission Spectroscopy (XPS), X-ray Diffraction (XRD), Diffuse Reflectance Spectra (DRS), Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray (EDX), FT-IR spectroscopy techniques…The adsorbent and photo-catalytic activities of the materials were estimated from the adsorption and degradation of phenol or methylene blue in their aqueous solutions.

Phosphate binders were studied to create the photo-catalytic TiO_2/HAp coatings on some surfaces. The photo-catalytic activity of TiO_2/HAp coating was also studied on the degradation of methylene blue (MB) in aqueous solution.

Main results:

TiO_2 prepared from Ilmenite ore after annealing at 750°C in 2 hours was in the form of anatase phase, with the average particle size (TEM) of around 10nm, and Eg of 3.21eV. TiO_2 Millennium after annealing at the same condition was also in the form of anatase phase, with the average powder size (SEM) of around 25÷35 nm, and Eg of 3.33eV. The HAp precipitated (precipitated HAp) at pH ≥ 9 and annealed at 750°C was of hydroxyapatite phase ($Ca_{10}(PO_4)_6(OH)_2$), rod-shaped powders, the crystalline percentage of 60%, and more OH groups on the surfaces. Meanwhile, the HAp prepared by hydrothermal method (hydrothermal HAp) and annealed at 750°C was of hydroxyapatite phase, but plate-leaf-shaped powders and E_g of 5.5eV.

TiO_2/HAp prepared by the precipitation method (precipitated TiO_2/HAp) had both anatase and hydroxyapatite phases. However, the product prepared by the hydrothermal method (hydrothermal TiO_2/HAp) included monetite phase (dicalcium phosphate anhydrous, DCPA, $CaHPO_4$). The precipitated TiO_2/HAp had the same band gap value as that of TiO_2 anatase. Meanwhile, the band gap values of the hydrothermal TiO_2/HAp were a little higher. The specific surface area of the precipitated TiO_2/HAp was higher than that of TiO_2 and pure HAp after annealing at the same temperature. The pore size values of these composites were equivalent to that of pure HAp but higher than that of pure TiO_2. The TiO_2/HAp material containing 90 wt% TiO_2 had the highest photo-catalytic activity in the degradation of phenol and MB in aqueous solution.

At 550°C, the zinc phosphate binder was converted from colloidal $Zn(H_2PO_4)_2$by the following diagram: $Zn(H_2PO_4)_2 \rightarrow Zn_4(PO_4)_2(OH)_{2.}\ 3(H_2O) \rightarrow Zn_2P_2O_7$. Whereas, the aluminum phosphate binder was converted from colloidal $Al(H_2PO_4)_3$ into $AlPO_4$. In the same conditions, the coating with colloidal aluminum phosphate gains a better adhesion than the coating with zinc phosphate. By increasing the content of TiO_2/HAp powders in the mixture, the photo-catalytic activity of TiO_2/HAp coating increased and reached the maximum at the TiO_2/HAp content of 12% (wt). The photo-catalytic activity of the coatings had been shown both in the solution and in the air environments.

Based on the results obtained in this study, it can be concluded that the adsorbent and photo-catalytic activity of TiO_2/HAp material containing the proper HAp percentage is higher than that of pure TiO_2. To improve the application of the photo-catalytic TiO_2/HAp material, the photo-catalytic coatings can be created by using aluminum phosphate binders. Additionally, Ilmenite ore can be used as the original material to prepare the photo-catalytic TiO_2/HAp material.

Advisors	**PhD. Student**
Assoc. Prof. Dr. Phan Dinh Tuan	Nguyen Thi Truc Linh
Dr. Nguyen Van Dung	