

## CHAPTER V

### CONCLUSIONS

The synthesis of nanocrystalline  $\text{TiO}_2$  and the deposition of  $\text{TiO}_2$  on  $\text{Al}_2\text{O}_3$  bead and glass tube have been demonstrated. The sol containing about 4 nm  $\text{TiO}_2$  crystals was prepared by an alcohol-based sol-gel method from tetrabutyl orthotitanate in the presence of a small amount of water and acid catalyst. Phase composition, crystal microstructure and physical property of the obtained powder were found to be strongly dependent on the synthesis conditions such as concentration of the Ti precursor, water content, aging time, as well as type of catalyst.

It was found that more concentrated Ti solution led to faster sol formation and greater yield of the  $\text{TiO}_2$  particles. Increasing water content caused faster hydrolysis which resulted in a rapid formation of the  $\text{TiO}_2$  sol. High water content was also led to bulk precipitation instead of forming the stable sol, and was found to promote anatase-to-rutile phase transformation. In addition, this phase transformation as well as crystallite growth underwent upon aging. By comparison HCl and  $\text{HNO}_3$  catalysts, HCl catalyst promoted the formation of rutile phase while  $\text{HNO}_3$  catalyst promoted the formation of the anatase phase. By adding sufficient amount of  $\text{HNO}_3$  into the precursor solution, pure anatase was obtained.

By performing various calcination conditions on pure anatase, physical characteristics of the  $\text{TiO}_2$  powders such as phase composition, crystallite size and specific surface area were manipulated. Depending on calcination temperature and time, anatase-to-rutile phase ratio was varied in the entire range, *i.e.*, from pure anatase to pure rutile. The crystallite sizes of the anatase and rutile were ranged from ~4 – 35 nm and ~20 – 60 nm, respectively. The highest specific surface area of ~240  $\text{m}^2/\text{g}$  was obtained on the as-synthesized powder, and reduced to ~6  $\text{m}^2/\text{g}$  after calcination at 700°C for 4 h. The lowest

temperature for anatase-to-rutile transformation was observed at around 300°C, which was relatively low. Low phase transition temperature may restrict its application to only moderate temperature. It is the subject of future work to bring it to higher temperature.

Photocatalytic efficiency of the TiO<sub>2</sub> powder towards Cibracon red was dependent on phase composition and physical property which were altered by calcinations. The powders containing pure anatase and possessing very fine crystal size and large specific surface area were the most photocatalytically reactive which were comparable to that of the commercial P-25. Mixed anatase and rutile phases were less active, while pure rutile was the least active. Incorporation of vanadium into the TiO<sub>2</sub> did not improve photocatalytic efficiency. It was believed that the vanadium was present in vanadium oxide form which was photocatalytically inactive.

The sol-gel derived TiO<sub>2</sub> was successfully deposited onto the Al<sub>2</sub>O<sub>3</sub> bead and glass tube by dip coating and spraying, respectively. The TiO<sub>2</sub> particles uniformly deposited onto the surface as well as inside the open pores of the bead. Likewise, by manipulating the spraying procedure, uniform TiO<sub>2</sub> coating was applied onto the glass tube surface. In both cases, the amount of TiO<sub>2</sub> loading or coating thickness was monitored by controlling number of dipping or spraying cycle. The photocatalytic efficiency of TiO<sub>2</sub> coating on Al<sub>2</sub>O<sub>3</sub> bead was lower than that of the TiO<sub>2</sub> powder. By comparison to the TiO<sub>2</sub> suspension, some of the TiO<sub>2</sub> particles deposited inside the pore and at the Al<sub>2</sub>O<sub>3</sub> surface were blocked from the UV light by the surrounding Al<sub>2</sub>O<sub>3</sub> bead and therefore resulted in lower efficiency compared to that of the TiO<sub>2</sub> powders. Finally, the TiO<sub>2</sub> coating on the glass tube was able to oxidize methanol under UV illumination. The efficiency was improved by using thicker TiO<sub>2</sub> coating.