

# CHAPTER I

## INTRODUCTION



### 1.1 Motivation

Titanium dioxide ( $\text{TiO}_2$ ) is one of the most interesting nanostructured materials at the present time, since it possess superb catalytic properties and excellent stability after chemical reaction. It is generally used in industry as a photocatalyst. Properties effecting the photocatalytic activity of  $\text{TiO}_2$  particles have been reported to include surface area, crystallite size and crystal structure (Ding and Liu, 1997; Salim et al., 2000). Consequently, the manipulation of microstructure, especially for nanocrystalline powders, is crucial to optimize photocatalytic efficiency in order to apply this nanosized particle in contaminant removal using chemical reaction in environmental application. For  $\text{TiO}_2$ , the mainly polymorphs among others are anatase and rutile. It is generally accepted that anatase is the most active photocatalyst due to its wider bandgap than rutile (3.23 eV for anatase and 3.1 eV for rutile), and that a combination of high crystallinity and large specific surface area improves photocatalytic performance (Moon et al., 2001; Ovenstone, 2001). These physical properties are controlled by the preparation method.

There are many methods to synthesize  $\text{TiO}_2$  nanoparticles, such as chemical vapor deposition (CVD) (Ayllon et al., 1999), the sol-gel technique (Poniatowski et al., 1994), hydrolysis (Park et al., 1997) and the microemulsion method (Kim and Hahn, 2001). Sol-gel technique is the most common method of producing  $\text{TiO}_2$  nanoparticles and it is accepted to be the practical process to control the structure of a material on a nanometer scale (Hench and West, 1990)

This study was focused on preparation and characterization of  $\text{TiO}_2$  nanoparticles using sol-gel method for photocatalysis process in treating heavy metals from industrial wastewater. To ensure that the synthesized nanoparticles can be used efficiently in heavy metals removal, an evaluating of performance and a determination of photocatalytic activity in selected heavy metal removal was also investigated.

In this work, the selected contaminant was chromium (VI), the most toxic specie and frequently found heavy metal in industrial waste streams. Chromium (VI)

has been classified as the most employed heavy metal in many industrial processes in Thailand. Therefore, the appearance of this heavy metal in the improper discharge of effluent streams from chrome plating, electronic, timber and leather tanning industries could be found. These effluents must be treated and the chromium (VI) concentration must be removed from the waste stream according to its relatively high toxicity and mobility. It was reported that chromium (VI) has been shown to be carcinogenic by inhalation and is corrosive to tissue (Fu et al., 1998). The United Nations Food and Agriculture Organization recommended maximum level of this heavy metal for irrigation water is 100 $\mu$ g/L. The limitation of chromium (VI) in treated effluent 0.25 mg/L is regulated by the Pollution Control Department, Thailand.

In general, the practical way to remove chromium (VI) is a photocatalytic process (Kajitvichyanukul, 2002; Kajitvichyanukul, 2003). The commercial TiO<sub>2</sub> powder used in the previous work exhibited the limitation in the active surface area, and the improper crystallinity as high amount of rutile presented in crystal structure. Thus, aim of this work was to synthesize the novel nanoparticle TiO<sub>2</sub> with superb properties for better efficiency in chromium (VI) removal using photocatalysis process as indicated above.

## 1.2 Objectives

Major objective of this work was to synthesize and characterize a novel nanoparticle TiO<sub>2</sub> with proper properties (high surface area, small nanocrystal size, high porosity, and proper crystal structure) and high photocatalytic activity in chromium (VI) removal application.

Minor objectives in this study included:

- to investigate the effect of stabilizing agents on properties of nanoparticle TiO<sub>2</sub>
- to investigate the effect of molar ratios of TiO<sub>2</sub> to stabilizing agent on properties of nanoparticle TiO<sub>2</sub>
- to investigate the effect of calcination temperatures on properties of nanoparticle TiO<sub>2</sub>

### 1.3 Hypotheses

- Photocatalysis efficiency in chromium (VI) removal using nanoparticles  $\text{TiO}_2$  was controlled by  $\text{TiO}_2$  properties.
- Stabilizing agents: diethylene glycol (DEG) and polyethylene glycol (PEG) changed the properties of  $\text{TiO}_2$ , which lead to either increasing or decreasing process efficiency.
- Mole ratios of TTiP to stabilizing agent and calcination temperatures were the major factors controlling  $\text{TiO}_2$  properties in preparation of nanoparticle  $\text{TiO}_2$ .

### 1.4 Scopes of the Study

All experiments in this research were conducted on the laboratory scale. The scope of this work was as follows:

1. Nanoparticles  $\text{TiO}_2$  will be prepared by sol-gel technique.
2. Two stabilizing agents: DEG and PEG, will be used to control the properties of nanoparticle  $\text{TiO}_2$ .
3. Characteristics of nanoparticles  $\text{TiO}_2$  studied are based on photocatalysis purposes only. Major properties to be studied include surface area, porosity, pore volume, and ratio of anatase to rutile.
4. Photocatalytic activity of nanoparticles  $\text{TiO}_2$  will be measured using chromium (IV) in synthesis wastewater with the concentration 50 mg/L, which corresponds to the real wastewater from the studied industry. The mechanisms of the chromium (VI) photoreduction will not be included in this study.

### 1.5 Expected Outcome

- Obtain nanoparticle  $\text{TiO}_2$  with novel properties beneficial to the photoactivity.
- Gain a better understanding of the role of types and amount of stabilizing agents on properties of nanoparticle  $\text{TiO}_2$ .
- Gain a better understanding of the effect of molar ratios of  $\text{TiO}_2$  to stabilizing agent on properties of nanoparticle  $\text{TiO}_2$ .
- Gain a better understanding of the effect of calcinations temperatures on properties of nanoparticle  $\text{TiO}_2$ .