

OPTIMIZATION OF TITANIUM EXTRACTION FROM DRINKING WATER
TREATMENT PLANT RESIDUE AS POTENTIAL PHOTOCATALYST

NOR SYAHIDAH BINTI ZULKAPLI

UNIVERSITI TEKNOLOGI MALAYSIA

OPTIMIZATION OF TITANIUM RECOVERY FROM DRINKING WATER
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NOR SYAHIDAH BINTI ZULKAPLI

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To my beloved parents..

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In the name of Allah, the Most Beneficent, the Most Merciful

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ABSTRACT

Titanium dioxide (TiO_2) is known for its excellent photocatalytic activity and many industries have now shifted their efforts to recover TiO_2 from secondary sources. Hydrometallurgy techniques which comprise of leaching, purification and precipitation have been commonly used to recover TiO_2 . However, this technique involves many processes and further work is needed to improve the laboratory process. This study aims to optimize the titanium leaching from drinking water treatment plant (DWTP) residue using response surface methodology (RSM). The parameters studied were acid concentration, temperature and solid to liquid ratio. The purification work was carried out using solvent extraction and the percentage recovery of titanium using Cyanex 272 and Cyanex 301 were investigated. The final product of TiO_2 was obtained via precipitation using magnesium oxide. The performance of TiO_2 as photocatalyst was evaluated using methylene blue and fungi under UV light irradiation. From the characterization study, it was found that the DWTP residue was poorly crystalline and predominantly consists of kaolinite and quartz with minor constituent of hematite, illite and anatase. Experimental results showed that a maximum leaching of titanium was obtained at 66% with optimum condition were found to be at 5.5 M acid concentration, 62°C heating temperature and 10 g/L solid to liquid ratio. In solvent extraction, the best titanium recovery was achieved at 86% using Cyanex 272 with low extraction of aluminium and iron. In contrast, Cyanex 301 showed 60% titanium recovery with 54% of aluminium was extracted into the organic phase. The X-ray Fluorescence (XRF) analysis indicated that 71% of TiO_2 was produced. In addition, X-ray Diffraction (XRD) confirmed the formation of TiO_2 with the crystalline anatase phase detected at 2θ values of 25.3°, 37.8°, 48.0°. FESEM micrograph of TiO_2 showed that the aggregates were present in the form of uniform spherical shape with considerable variation of particle size. The photocatalytic activity of the final product TiO_2 under UV light irradiation showed maximum degradation (84%) with 0.5 g/L TiO_2 loaded in methylene blue solution. At similar amount of loaded TiO_2 , the findings indicated that almost 50% of the fungi growth was inhibited within 14 days. Additionally, zero inhibition of fungi growth was observed without the presence of TiO_2 . In summary, the photocatalyst TiO_2 had been successfully recovered from DWTP residue using an optimized leaching process, hence contributes to the improvement of hydrometallurgy technique.

ABSTRAK

Titanium dioksida (TiO_2) dikenali antara pemangkin yang terbaik dan kebanyakan industri kini berusaha untuk mendapatkan TiO_2 daripada sumber sekunder. Teknik hidrometalurgi yang terdiri daripada pelarutan, purifikasi dan pemendakan kebiasaannya digunakan bagi mendapatkan TiO_2 . Walau bagaimanapun, teknik ini melibatkan proses yang banyak dan kajian lanjutan diperlukan bagi meningkatkan kualiti kerja makmal. Kajian ini bertujuan untuk mengoptimumkan proses pelarutan titanium daripada bahan baki loji rawatan air minuman (DWTP) dengan menggunakan kaedah gerak balas permukaan (RSM). Parameter yang dikaji ialah kepekatan asid, suhu dan nisbah pepejal kepada cecair. Proses penulenan telah dilakukan dengan menggunakan kaedah pengekstrakan pelarut dan kadar peratusan pengekstrakan titanium telah diselidik menggunakan bahan organik iaitu Cyanex 272 dan Cyanex 301. Hasil akhir TiO_2 telah diperolehi melalui kaedah pemendakan yang menggunakan magnesium oksida. Penilaian produk TiO_2 sebagai bahan pemangkin telah dijalankan dengan menggunakan metilena biru dan kulat di bawah sinaran cahaya UV. Hasil kajian perincian mendapati bahan baki DWTP mempunyai struktur kristal yang lemah dan kebanyakannya terdiri daripada kaolinit dan kuarza dengan sebilangan kecil bijih besi, ilit dan anatase. Hasil eksperimen menunjukkan bahawa pengekstrakan titanium yang maksimum telah diperolehi sebanyak 66% pada keadaan optimum dengan kepekatan asid 5.5 M, suhu pemanasan 62°C dan nisbah pepejal kepada cecair 10 g/L. Dalam pengekstrakan pelarut, 86% titanium telah diekstrak menggunakan Cyanex 272 dengan peratusan aluminium dan ferum yang rendah. Hasil perbandingan menunjukkan Cyanex 301 telah mengekstrak titanium sebanyak 60% dan aluminium yang turut diekstrak dalam fasa organik adalah 54%. Analisis X-ray Fluorescence (XRF) menunjukkan sebanyak 71% TiO_2 telah dihasilkan. Pembelauan X-ray (XRD) telah membuktikan pembentukan fasa berhablur anatase pada nilai 2θ adalah 25.3° , 37.8° , 48.0° . Mikrograf FESEM menunjukkan gumpalan TiO_2 berbentuk sfera yang tidak sekata dengan pelbagai saiz zarah. Proses pemangkinan di bawah sinaran UV menggunakan produk TiO_2 menunjukkan penguraian maksimum (87%) apabila 0.5 g/L TiO_2 dimasukkan dalam larutan metilena biru. Pada jumlah yang sama, hasil eksperimen menunjukkan bahawa hampir 50% daripada pertumbuhan kulat telah dihalang dalam tempoh 14 hari. Pertumbuhan kulat secara maksimum telah diperhatikan apabila tiada kehadiran TiO_2 . Secara ringkas, bahan pemangkin TiO_2 telah berjaya dihasilkan daripada bahan baki DWTP dengan menggunakan kaedah pelarutan yang telah dioptimumkan, dan sekaligus menambah baik kaedah hidrometalurgi.

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LIST OF ABBREVIATIONS

DWTP	Drinking water treatment plant
TiO ₂	Titanium Dioxide
RSM	Response Surface Methodology
CCD	Central Composite Design
ANOVA	Analysis of Variance
XRF	X-ray Fluorescence
XRD	X-ray Diffraction
ICP	Inductively Coupled Plasma
FTIR	Fourier Transform Infrared
UV	Ultraviolet
FESEM	Field Emission Scanning Electron Microscopy
g/L	Gram/litre
SX	Solvent Extraction
MB	Methylene Blue
PDA	Potato Dextrose Agar

CHAPTER 1

INTRODUCTION

1.1 Background of study

Titanium dioxide (TiO₂) is an oxide of titanium that occurs naturally and it is proven to be effective for its catalytic properties. Research on photocatalytic degradation of organic pollutants and microorganism has been in a continuous expansion in the recent decades and TiO₂ is still by far the most used photocatalyst (Fresno *et al.*, 2014). The use of semiconductor photocatalyst have shown to be advantageous and useful in the treatment of waste water pollutants such as methylene blue because of its high stability in biological and chemical environment (Mohapatra and Parida, 2006; Arbuj *et al.*, 2010). In addition, the biodeterioration of aesthetic and structural damage of construction materials is prominently caused by the presence of numerous microbial cells, particularly tangles of fungal hyphae, as well as dust. As a consequence, there is a need to develop an antimicrobial coating that employs the application of TiO₂ photocatalytic reactions to protect the external building elements from the bio-deterioration of microorganisms, especially fungi.

Current emphasis on industrialization and rapid growth of technology has led to a high demand of titanium. Due to the increment of titanium consumption, the primary source of titanium (rutile) is reported to become scarce (Zhang *et al.*, 2011a). As a consequence, most of the titanium dioxide (TiO₂) are synthesized using various physical and chemical methods. Some of the commonly used synthetic methods to produce TiO₂ are chemical vapor deposition (CVD) (Karaman *et al.*, 2013), oxidation of titanium tetrachloride (Wang *et al.*, 2012), sol gel technique

(Blanco *et al.*, 2015; Leyva-Porras *et al.*, 2015; Pazokifard *et al.*, 2015) and thermal decomposition of titanium alkoxides (Li *et al.*, 2015). The synthesizing of TiO₂ using the available methods are often involves rigorous reaction, high energy consumption and potentially hazardous.

Alternatively, the recovery of titanium from secondary sources is preferable as this would unlock large tonnage of remaining raw sludge as feed material for production of desired metal oxide. Since decades ago, many studies were conducted on the recovery of titanium from various kind of secondary sources for instance blast furnace slag (Zhang *et al.*, 2007), red mud (Agatzini-Leonardou *et al.*, 2008), sand beach (Begum *et al.*, 2012), titanium slag (Liu *et al.*, 2013) and submerged-arc welding slag (Annoni *et al.*, 2013).

On a global scale, available literature estimates that 10, 000 tons of drinking water treatment plant (DWTP) residue is produced daily in most municipalities worldwide and directly disposed to a landfill (Babatunde and Zhao, 2006). To date, it has been widely used in the buildings and construction materials such as cement (Rodríguez *et al.*, 2010) and ceramics (Teixeira *et al.*, 2011). DWTP residue is mainly consists of clay, fine silt and sediment which are primarily composed of Si, Ti, Al, Fe, Mg, Ca, Na and K (Tour, 1989). Titanium is among one of the most valuable components found in the residue and therefore it can be used as secondary raw material for recovery of titanium.

The previous literatures stated that the method for metal extraction is classified into pyro-metallurgical, hydro-metallurgical and bio-metallurgical. The pyro-metallurgical extraction of titanium normally involved smelting, roasting and refining (Lasheen, 2008). However, this process has been criticised for many years due to high emission of carbon footprint. The bio-metallurgical process involving bacterial leaching has been reported to give poor recovery of titanium (Jonglertjanya and Rubcumintara, 2013). Hydrometallurgical process involves leaching via chemical method and further purifications technique such as solvent extraction is required to concentrate the metals (Borsalani *et al.*, 2011; Swamidoss and Malkhede, 2014).

Acid extraction has been acknowledged as an efficient method in metal recovery as most of the inorganic constituent including titanium are destabilized at low pH range. Direct acid leaching may also offer high solubility of metal complex and high leaching rates. The extraction of various metals including zinc (Oustadakis *et al.*, 2010), chromium (Jiang *et al.*, 2014), aluminium (Shemi *et al.*, 2014; Nayak and Panda, 2010), cobalt (Liu *et al.*, 2015), among others, were successfully performed using sulphuric acid.

It is crucial to determine the most significant variable and interaction between two variables in an experiment which can be achieved using a statistical design analysis (Somasundaram *et al.*, 2014). Nowadays, the uses of Response Surface Methodology (RSM) has been studied extensively mainly on the metal recovery including copper (Somasundaram *et al.*, 2014), aluminium (Shemi *et al.*, 2014), zinc (Zhang *et al.*, 2010), chromium (Sahu *et al.*, 2009), cadmium (Iqbal *et al.*, 2016), vanadium and nickel (Nazari *et al.*, 2014), manganese (Azizi *et al.*, 2012) and gold (Ha *et al.*, 2014). In this study, the application of RSM to design an optimum hydrometallurgical technique is aimed at maximum recovery of titanium from DWTP residue.

1.2 Problem statement

The hydrometallurgical technique is chosen as it provides several substantial advantages including the ability to control the level of impurities, high potentials for metal recovery and suitability for small scale application. However, this technique is divided into many levels of processes which is leaching, purification that involves solvent extraction with stripping and finally precipitation process. This consequently prolonged the experimental work, making it laborious and difficulty to reach the optimal conditions due to the fact that the interaction between variables is ignored. Among the possible approaches, a statistical design method of RSM is found to be practical. This method could reduce a number of experiments to be done in the lab work where this method will be suggesting the best experimental condition based on

the design model. In this study, the optimization of hydrometallurgical technique mainly on the leaching part is highlighted.

1.3 Objectives of the study

The optimization of the hydrometallurgy technique to recover TiO_2 from DWTP residue is divided into first and second objective. The third objective refers to the photocatalytic application of the recovered TiO_2 . The objectives of the research are simplified as follows:

- i) To optimize the parameters affecting leaching of titanium which include acid concentration, temperature and solid to liquid ratio using RSM
- ii) To investigate on the types of organic extractants on the recovery of titanium.
- iii) To evaluate the photocatalytic activity of recovered TiO_2 against methylene blue and *F. equiseti* under UV light irradiation.

1.4 Scope of the study

This part is discussed according to the objectives; (i) optimization of leaching process, (ii) purification process using solvent extraction and (iii) photocatalytic activity of recovered TiO_2 .

In the first objective, three independent variables namely acid concentration, temperature and solid to liquid ratio were optimized using central composite design (CCD) of RSM. The concentration of metals in the leached solution was determined using Inductively Coupled Plasma – Optical Emission Spectrometer (ICP-OES). The chemical composition and properties of DWTP residue and leaching by-product were analyzed using X-ray Fluorescence (XRF) spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy, X-ray Diffraction (XRD) spectroscopy, Scanning Electron Microscope (SEM) and Field Emission Scanning Electron Microscope (FESEM).

For second objective, the purification of sulphate leached solution was conducted using solvent extraction. Two types of selective anionic extractants (Cyanex 272 and Cyanex 301) were evaluated. The stripping of titanium was performed using HCl and final TiO_2 was obtained via precipitation with light magnesium oxide. The produced TiO_2 was calcined at 550°C and further characterized using XRF, XRD and FESEM analysis.

In the third objective, the photocatalytic activity of TiO_2 was evaluated by the degradation of the organic dye pollutant, methylene blue (MB) in water under UV light irradiation. The amount of TiO_2 loaded into the MB solution was varied and the degradation rate of MB was observed within four hours under UV irradiation. Then, similar amount of TiO_2 loaded obtained from the photocatalytic activity of MB was used against isolated fungi. Fungi sample was collected at C07 UTM exterior building and the fungi sample was identified using polymerase chain reaction (PCR). The efficacy of TiO_2 against fungi was observed at the time intervals of 1, 7, and 14 days by measuring the diameter of fungal colonies.

1.5 Significance of the study

The main aim of this study is to recover TiO_2 from DWTP residue using a hydrometallurgical technique. Optimization of this technique is necessary as it can reduce the laborious work and for more feasible metal recovery in full-scale applications. This study will also contribute as a promising technique for the recovery of titanium from different kind of sources. Apart from that, product of this research which is TiO_2 can be further utilized as a photocatalyst material. Photocatalytic application can provide solutions for many of the environmental challenges as most of the organic pollutants will undergo degradation to produce less harmful or non-harmful substances under UV light irradiation.

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