UNIVERSITI TEKNOLOGI MARA

PREPARATION AND OPTIMIZATION OF SELF-ORGANIZED TiO_2 NANOTUBES FORMATION BY ANODIC OXIDATION

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AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the result of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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ABSTRACT

The main focus of this research is to synthesize and to optimize the properties of TiO_2 nanotube with its final applications as photocatalyst for degradation of dye water. The growth mechanism of self-organized TiO_2 nanotube formation that were prepared by anodization of Ti foil in an electrolyte containing F^- ions were also proposed in this work. Various parameters were conducted and analyzed. These included the different voltage, different anodization time, different pH, different electrolytes, and different annealing temperature. Since the morphological and structural properties were the main data that needed in order to understand the mechanism of TiO_2 nanotube formations, therefore several characterization technique were used such as field emission scanning electron microscope (FESEM) with EDX for morphological and elemental studies, X-ray diffraction spectrometer (XRD) for structural and phase identification, and UV-VIS for optical study. The optimum parameters were finally gathered with well-organized and uniform distribution of TiO_2 nanotubes which were formed at 19 V for 30 min in pH 3 electrolyte which contained 0.7g NH_4F and 100 ml $NaSO_4$. FESEM results, showed that the diameter of nanotubes changed directly proportional to the parameter control. For example as the voltage increased the diameter were increased. In here, the smallest diameter achieved was at 30 nm and the biggest diameter achieved was at 100 nm. The length of TiO_2 nanotube were also changed and the highest length achieved was at 500 nm. However, FESEM images clearly shown that nanotubes were hollow in shape with mouth opening at the top end and closed at the bottom of the tubes. The EDX analysis showed that Ti, and O, elements were existed in the sample. Thus proved that TiO_2 nanotube were occurred. The XRD results showed that, for pure Ti foil and as-anodized sample only appeared strong peak of Ti and weak amorphous peak of TiO_2. However, the crystalline sharp peak were appeared at 400 °C to 800 °C. Whereby, the phase was transformed from anatase to mixture of anatase and rutile from at 500 °C. The TiO_2 nanotubes had shown a good result in photocatalytic activity under degradation of mixture of anatase and rutile (500 °C) at 19 V and pH 3 within 1 hour of methyl red degradation. TiO_2 nanotubes with thinner walls and longer tubes have a more pronounced photocatalytic activity as compared with non-uniform thicker wall and longer. The schematic diagram and detail discussion on growth mechanism were explained in Chapter 4.

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CHAPTER ONE

INTRODUCTION

1.1 RESEARCH BACKGROUND

Titanium dioxide (TiO_2) is an important inorganic functional material that has been widely investigated among other metal oxide, due to its physical and chemical properties that contribute to innumerable applications such as biocompatibility (Roy et al., 2011; Liu et al., 2011), self-cleaning (Wang et al., 1997), and photocatalyst (Dong et al., 2014; Chong et al., 2013; Shou et al., 2012). The physical structure of TiO_2 in nanosized structure plays a crucial role in the activation or enhancement of TiO_2 as photocatalyst in various applications as mentioned before. This is known as one dimensional (1D) nanostructure materials, such as nanowire (Ablat et al., 2014), nanorod (Ohno et al., 2014), and nanotubes (Bessegato et al., 2014).

Recently, numerous low-dimensional TiO_2 materials such as nanowires, nanotubes and nanorods were synthesized successively (Yuan and Su, 2004). Self-organized nanotubular oxide structures were produced more on a range of transition metals, such as zinc, titanium and vanadium (Tsuchiya et al., 2004), and also alloys such as copper and brass that have attracted much attention due to their large surface area (Zhang et al., 2014), excellent charge-transport properties (Zhang et al., 2010), ease of handling, excellent mechanical properties, high corrosion resistance, and good biocompability (Rautray et al., 2011; Xi et al., 2006). Properties of excellent charge transport as TiO_2 is active only under ultraviolet (UV) light because of its wide band gap (~3.0 eV). Other properties are due to its unique optoelectronic and photochemical properties, high refractive index, high dielectric constant and excellent optical transmittance.

While anodization of Ti has been extensively investigated in various electrolytes, particularly acidic electrolytes containing Cl^- (Sul et al., 2002) and Br^- ions (Virtanen and Curty, 2004), only a small number of reports regarding anodization of Ti in HF^- or F^- containing electrolytes have been published until this decade (Zwilling et al 1999; Chazalviel et al., 2000; Schultze et al., 2000).