UNIVERSITI TEKNOLOGI MARA

MODIFICATION AND CHARACTERIZATION OF MICROWAVE ASSISTED INTERSTITIAL N-DOPED TiO_2 FOR PHOTODEGRADATION OF RR4 DYE

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ABSTRACT

Mostly, researchers were prepared nitrogen doped titanium dioxide (N-doped TiO_2) using chemical preparation such as solvothermal, ion implantation, sol-gel, calcination, sonochemical and sputtering technique and this method are very complicated due to the process either using specific equipment or consumed chemical to dissolved the precursor. The preparations N-doped TiO_2 under microwave treatment was also conducted using sol-gel method. Fewer works exploited by using solid state method and most of them was prepared N-doped TiO_2 under muffle furnace. Therefore, no study focused on the modification N-doped TiO_2 using commercially available TiO_2 under microwave. Thus, an interstitial N-doped TiO_2 has been synthesized under microwave irradiation using urea as nitrogen precursor and commercially available P25-TiO_2. The preparation N-doped TiO_2 was undergoes solid state method with contributed to the formation of interstitial, which is in contrast to other solid state methods that give substitutional N-doped TiO_2. The various ratio of TiO_2 and urea was mixed using mechanical mixing and heating under microwave irradiation using various irradiation power. The optimization study was applied under photodegradation of Reactive Red 4 (RR4) dye for 50 min under 55-W fluorescent lamp. The comparison study was done by comparing with different irradiation light (visible and solar irradiation) and comparing with immobilization technique using Double Sided Adhesive Tape (DSAT) technique.The optimum modified N-doped TiO_2 found at 3 g urea in 7 g TiO_2 denoted as U3-800 prepared at 800 W of microwave irradiation under 30 min constant time. It was observed that N has chemically bonded with titania producing interstitial Ti-O-N proven by X-ray Photoelectron Spectroscopy (XPS) analysis at 400.3 and 404.8 eV. The interstitial Ti-O-N also can be exposed by Fourier Transform Infrared Spectroscopy (FTIR) suggested as hyponitrite formation. There is no transformation phase occurred in U3-800 compared with unmodified TiO_2 in X-ray diffraction (XRD) analysis. The band gap energy (Eg) of the modified sample was *ca.* 2.9 eV as detected by UV/Vis-DRS. Photodegradation rate of RR4 dye under U3-800 is 1.6 times faster in comparison with unmodified TiO_2 as well as control TiO_2 under a normal 55-W fluorescent lamp. An active photo response for U3-800 sample under visible light irradiation observed with 80 min of time to complete RR4 color removal while no photocatalytic degradation detected from unmodified and control TiO_2. Immobilization of the U3-800 sample was applied by using double sided adhesive tape (DSAT) as a thin layer binder and the photoactivity of immobilized U3-800 is comparable to the suspension mode. The immobilized U3-800 is durable and can continuously used for 30 cycles without affecting the photoactivity performance. This simple technique will make ease to industrial to prepare the modified TiO_2 and help the pre-treatment of industrial to degraded the pollutant in twenty-four hour. Moreover, this physical technique prepared N-doped TiO_2 was also promising the green technology and environmental friendly which successfully producing interstitial type N without using any solvent.

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CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND OF STUDY

Dyes or pigments are widely used in the industries like textile, paper, cosmetic and plastic for coloring of their products. Excessive usage of dyes will turn to the dye product waste which is difficult to degrade since most of dyes contain highly carcinogenic aromatic amine (Cristina *et al.*, 2010). Ogugbue and Sawidis (2011) have reported that the textile industries was estimated discharged over 200,000 tons every year in dyeing and finishing operation. Most of these dyes escaped from the conventional wastewater treatment processes and persist in the environment (Couto, 2009).

The treatment of dye wastewater has received considerable intentions since most of dyes are highly carcinogenic, mutagenic, and toxic. It is probably will decrease the light to penetrate during photosynthesis. Moreover, the dye wastewater will cause the oxygen deficiency, aquatic life failure and limiting beneficial uses of water bodies for human such as for recreation, drinking water and irrigation. Some of the wastewater treatment such as adsorption, membrane, chemical and biological treatment becoming less efficient to complete decolorized. In the textile manufacturing wastewater treatment, they have used anti-microbial agent resistant to biological degrade using natural fiber such as cotton. Meanwhile, in wastewater industries have treated the municipal sewage treatment plants by using coagulation and flocculation process (Curtis *et al.*, 2005; Mohabansi *et al.*, 2011). However these techniques are less effective and transferred the non-biodegradable matter into sludge. Table 1.1 shows the description of current wastewater treatment processes.