

UNIVERSITI TEKNOLOGI MARA

**PHOTOCATALYTIC BEHAVIOUR
OF NIOBIUM-DOPED TITANIUM
DIOXIDE NANOPARTICULATE
FILMS FOR PHOTODEGRADATION
OF METHYLENE BLUE**

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MSc

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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 results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-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

TiO₂ nanostructures is one of the promising metal oxide semiconductors which has been used in many applications such as solar cells, sensors, photocatalysts, and even for medicines. Photocatalysis using nano-TiO₂ is an excellent approach in overcoming the water pollution problems. However, there are some issues regarding TiO₂ shortcomings in photocatalysis such as leaving behind residual by-products of TiO₂ powder suspension that needed an after treatment and limited light absorption in the visible region. Therefore, this current study was performed to synthesize TiO₂ photocatalyst on substrates and doping approach with niobium (Nb) was introduced to enhance the crystallinity and optical properties of TiO₂. In this work, TiO₂ nanoparticulate films have been synthesized on glass substrates at fixed molarity of 0.25 M. In this part, the annealing temperatures and coating cycles were varied from 400-600 °C and 1-9 cycles, respectively. Further, TiO₂ were doped with Nb where the dopant concentrations were manipulated from 0 (undoped) to 9 at.% and the annealing temperatures were varied from 400-600 °C. Photocatalytic behaviour Nb-doped TiO₂ nanoparticulate films for the photodegradation of Methylene Blue (MB) were also investigated. The structural, morphological and optical properties of the synthesized films were characterized via Field-Emission Scanning Electron Microscope (FESEM), X-ray diffraction (XRD) and ultraviolet-visible spectrophotometry (UV-Vis), respectively. The photocatalytic behavior of Nb-doped TiO₂ nanoparticulate films was also measured by using UV-Vis. Results show that the optimization of pristine TiO₂ nanoparticulate films at different annealing temperatures exhibited that the film annealed at 500 °C have the most uniform morphology, highly crystalline and has the highest absorptivity at $3.6 \times 10^7 \text{ m}^{-1}$ than the other samples. Meanwhile, the optimum coating cycles was 7 cycles owing to the film has the smallest nanoparticles size, smallest crystallite size at 13.4 nm and highest absorption at $3.7 \times 10^7 \text{ m}^{-1}$. Nb-doped TiO₂ nanoparticulate films were synthesized and their photocatalytic behavior was investigated at 5 ppm of MB concentration and neutral pH. 5 at.% of Nb-doped TiO₂ shows the highest absorption at $3.2 \times 10^7 \text{ m}^{-1}$ and the degradation of MB was also the highest at 95.9 % compared to the pristine TiO₂ film which only degraded up to 53.4 %. The optimized annealing temperature for Nb-doped TiO₂ nanoparticulate films was found to be at 550 °C and the percentage degradation of MB was 96.9 %. Further study on the effect of MB concentration and pH were investigated for the optimized Nb-doped TiO₂ films. It was found that 5 ppm of initial MB concentration has the highest degradation performance of 90.6 % and the optimum pH of MB was found to be at pH 9 where the percentage degradation was 98.3 %. These remarkable increases in photocatalytic behavior of Nb-doped TiO₂ nanoparticulate films were due to the increase in absorption where the absorbance was extended to the longer wavelength. Thus, Nb-doped TiO₂ nanoparticulate films are promising candidate in improving the photocatalytic activity for water purifications.

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