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SYNTHESIS AND CHARACTERIZATION OF ZINC OXIDE/GRAPHENE (ZnO/Gn) ON TITANIUM DIOXIDE/NIOBIUM PENTOXIDE (TiO_2/Nb_2O_5) THIN FILM FOR METHYLENE BLUE REMOVAL

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ABSTRACT

The existing challenges associated with the efficacy of TiO_2/ZnO thin films for photocatalytic wastewater treatment, including complex and costly seed layer coating methods, low photocatalytic efficiency, the removal of toxic compounds, and interfacial layer mismatch with ZnO and other elements, necessitate further investigation. This study aims to address these issues by depositing thin film samples of TiO_2-ZnO, optimising the parameters of graphene (Gn) (including atomic percentage, annealing, and immersion temperatures) in TiO_2-ZnO/Gn, and investigating the effects of incorporating 5 atomic percent of Nb_2O_5 into TiO_2-ZnO/Gn samples. In addition, it intends to assess the methylene blue removal on TiO_2-ZnO, TiO_2-ZnO/Gn, and TiO_2/Nb_2O_5-ZnO/Gn. Graphene-based materials excel in energy harvesting and water splitting, outperforming commercial standards in photocatalysis and treatment, thus, the addition of Gn helps to enhance methylene blue adsorption. Besides, it is added to reduced electron-hole pair recombination rate and band gap narrowing. Addition of 5 at. % Nb_2O_5 to the TiO_2-ZnO/Gn nanocomposite regulates electron behaviour, reducing recombination and improving photodegradation. The seed layer was applied to glass substrates through the sol-gel spin coating method, while ZnO/Gn was synthesised via the solution-immersion technique. The optimisation process involved varying Gn atomic percentages (11 at. %, 13 at. %, 15 at. %, 17 at. %, and 19 at. %), solution immersion temperatures (70ºC, 80ºC, and 90ºC), and annealing temperatures (400ºC, 450ºC, 500ºC, 550ºC, and 600ºC) for TiO_2-ZnO/Gn samples. 5 at. % Nb_2O_5 was introduced as a dopant in the TiO_2 seed layer and integrated into the optimised TiO_2-ZnO/Gn samples. Characterization techniques, including FESEM-EDX, XRD, HRTEM, and UV-Vis, were employed to assess morphological, structural, and optical properties. FESEM images confirmed the presence of Gn nanosheets and ZnO nanorods on the thin film, exhibiting distinctive grain-like structures in TiO_2/Nb_2O_5-ZnO/Gn. EDX analysis confirmed the materials present. X-ray diffraction analysis indicated crystallinity, with hexagonal wurtzite nanorods (prominent peaks at planes 100, 002, and 101), anatase TiO_2 (plans 101 and 200), Nb_2O_5 (plane 102), and Gn (plane 002) identified on the glass substrate. The HRTEM SAED patterns verified the crystal planes of the TiO_2, Nb_2O_5, ZnO, and Gn phases. The optimisation results identified 15 at.% Gn, 90°C solution immersion temperature, and 500°C annealing temperature as the optimised parameters for methylene blue removal. UV-Vis absorbance revealed that TiO_2/Nb_2O_5-ZnO/Gn exhibited the highest absorbance spectra, shifting towards visible light wavelengths. The energy band gap of TiO_2/Nb_2O_5-ZnO/Gn decreased from 3.0 eV to 2.6 eV. TiO_2/Nb_2O_5-ZnO/Gn demonstrated the highest degradation efficiency at 50.29%, outperforming TiO_2-ZnO/Gn (28.44%) and TiO_2-ZnO (29.66%). Collectively, TiO_2/Nb_2O_5-ZnO/Gn emerges as the most promising methylene blue removal.

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CHAPTER 1
INTRODUCTION

1.1 Research Background

The sun has been a great source of world illumination for a million years until today. It lightens the whole world by emitting UV and visible light rays. It is utilised to generate a few types of energy, such as photovoltaic and solar thermal systems. One of the main research projects in utilising solar energy is photocatalysis. Photocatalysis refers to reactions that occur when light and a semiconductor are interacted. A photocatalyst is a compound that absorbs light and functions as a catalyst for chemical reactions (Ameta et al., 2018). Because of its eco-friendliness, cost-effectiveness, ease of operation, and high efficiency, sun-driven catalysis has been demonstrated as a promising strategy for wastewater treatment (Charanpahari et al., 2019).

Meanwhile, wastewater pollution may pose a severe threat to human well-being. Pollution removal is a challenge to human society's sustainable development. For example, organic dye wastewater is produced in a significant amount as the textile sector expands, and the percentage of industrial wastewater shows an upward trend. These pollutants released into aquatic ecosystems had a significant impact on organism growth and survival, and some dyes with high toxicity and stability were unable to be degraded by microorganisms, resulting in a terrible ecological disaster (Wang et al., 2023). The semiconductor-based photocatalysis, which utilises solar energy, is regarded as a low-cost and attractive method for entirely converting pollutants from industrial release into harmless chemicals (Chenet al., 2020).

The fundamental idea of nanotechnology is the ability to produce nanoscale entities with significantly new features and molecular organisation (Barbara Karn et al., 2003). Nanomaterials have a greater surface-to-volume ratio than their conventional forms, which can increase their chemical reactivity and strength effect (Kundu et al., 2021).

In this study, the elements of TiO_2, Nb_2O_5, ZnO and Gn were mixed to form a novelty nanocomposite, to test for the methylene blue removal. A depth study of the synthesis of this nanocomposite towards forming the optimised photocatalyst was