GRAPHENE OXIDE-BASED HYBRID NANOCOMPOSITE FOR EFFICIENT PHOTODEGRADATION OF MALACHITE GREEN

ONG SUU WAN

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ABSTRACT

Photocatalyst attracted enormous attention due to its ability to fully degrade hazardous contaminants. This research work aimed to synthesis binary hybrid photocatalyst consists of iron (III) oxide (Fe_2O_3) and reduced graphene oxide (rGO): FG (1:1), FG (1:2), FG (2:1). The effect of polyaniline (PANI) addition was also studied in ternary hybrid photocatalyst: PFG (1:1), PFG (1:2), PFG (2:1). The synthesis process was carried out via oxidation method assisted by ultrasonication technique. The photocatalyst samples were characterised using X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Brunauer-Emmett-Teller (BET) surface area analysis, Field Emission Scanning Electron Microscopy (FESEM) with the EDX analysis, UV-Vis diffuse reflectance spectra (UV-DRS) and Photoluminescence Spectroscopy (PL). FTIR and XRD proved the presence of rGO and Fe_2O_3 in all FG photocatalysts while PFG photocatalysts have rGO, Fe_2O_3 and PANI. The FESEM analysis showed the distribution of Fe_2O_3 particles on the surface of rGO. The hybrid catalyst demonstrated a lower band gap energy and recombination rate, as evidenced by the UV-DRS and PL analyses, respectively. Photocatalytic capability of photocatalysts were evaluated through photodegradation of Malachite Green (MG) under ultraviolet light for 180 minutes. Optimization of the photodegradation performance were carried out by manipulating the reaction parameters such as dye concentration, photocatalyst dose and pH value. The finding showed FG (1:1) owned the highest degradation efficiency up to 89.2% at 60ppm dye concentration in pH 9. This is due to the synergic effect between Fe_2O_3 and rGO improved the active electron (e^-) and hole (h^+) generation and separation. In conclusion, FG (1:1) show a promising potential photocatalyst material for degradation of MG. This study implies on the exploration of the fundamental knowledge to develop a highly efficient photocatalyst for photodegradation of hazardous contaminants, particularly dyes in the application of wastewater treatment.

NANOKOMPOSIT HIBRID BERASASKAN GRAFIN OKSIDA UNTUK FOTODEGRADASI CEKAP BAGI MALAKIT HIJAU

ABSTRAK

Fotomangkin telah menarik banyak perhatian kerana keupayaannya untuk sepenuhnya mendegradasi bahan pencemar berbahaya. Kajian ini bertujuan untuk mensintesis fotomangkin kacukan binary yang teridiri daripada besi (III) oksida (Fe_2O_3) dan grafin oksida terturun (rGO): (FG (1:1), FG (1:2), FG (2:1)). Kesan penambahan polianalin (PANI) juga dikaji dalam pemangkin kacukan ternari. Proses sintesis dijalankan melalui kaedah pengoksidaan dibantu dengan teknik ultrasonik. Fotomangkin telah dicirikan menggunakan analisis belauan sinar-x (XRD), spektroskopi inframerah fourier transformasi (FTIR), analisis luas permukaan Brunauer-Emmett-Teller (BET), mikroskopi elektron penskanan pancaran medan (FESEM) dengan analisis EDX, spektroskopi UV-vis pantulan resapan (UV-vis DRS) dan Spektroskopi Fotopendarcahayaan (PL). FTIR dan XRD membuktikan kehadiran rGO dan Fe_2O_3 dalam semua fotomangkin FG manakala fotomangkin PFG mempunyai rGO, Fe_2O_3 dan PANI. Analisis FESEM menunjukkan taburan partikel Fe_2O_3 pada permukaan rGO. Pemangkin kacukan menunjukkan tenaga jurang jalur dan kadar penggabungan semula yang lebih rendah, melalui pembuktian oleh analisis UV-DRS dan PL, masing-masing. Keupayaan fotomangkin dinilai melalui fotodegradasi Malakit Hijau (MG) di bawah cahaya ultraungu selama 180 minit. Pengoptimuman prestasi degradasi dijalankan dengan memanipulasi parameter-parameter tindakbalas seperti kepekatan bahan pewarna, dos fotomangkin dan nilai pH. Dapatan menunjukkan FG (1:1) memiliki kecekapan degradasi Malakit Hijau tertinggi sehingga 89.2% pada kepekatan pewarna 60ppm dalam pH 9. Ini disebabkan kesan sinergik antara Fe_2O_3 dan rGO yang menambahbaik penghasilan dan pemisahan elektron (e^-) dan lubang (h^+) aktif. Kesimpulannya, FG (1:1) menunjukkan potensi tinggi sebagai bahan fotomangkin untuk degradasi MG. Kajian ini memberi implikasi kepada penerokaan terhadap ilmu asas untuk membangunkan fotomangkin hibrid yang berkecekapan tinggi bagi fotodegradasi bahan cemar berbahaya, khususnya bahan pewarna di dalam aplikasi rawatan air sisa.

.

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LIST OF ABBREVIATION

π-π	Stacking electrostatic
α	Alpha
A- Fe_2O_3	Hematite
γ- Fe_2O_3	maghemite
Au	Gold
Ag^+	Argentum ion
Ag_3PO_4	Silver phosphate
AOP	advanced oxidation processes
APS	Ammonium persulphate
BET	Brunauer-Emmett-Teller
CB	Conduction Band
CeO_2	Ceric oxide
CdS	Cadmium sulfide
ClO^-	Hypochlorite ion
ClO_2	Chlorine dioxide
CNT	Carbon nanotube
CO_2	Carbon Dioxide
COD	Chemical Oxygen Demand
e^-	Electron
EDX	Energy dispersive x-ray spectroscopy

eV	Electron Volt
Fe^{2+}	Iron (II) ion
Fe^{3+}	Iron (III) ion
Fe_2O_3	Iron (III) Oxide
Fe_3O_4	Magnetite, Iron (IV) Oxide
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier-transform infrared spectroscopy
GO	Graphene oxide
h^+	Proton
H^+	Hydrogen ion
H_2	Hydrogen
H_2O	Water
H_2O_2	Hydrogen peroxide
H_2SO_4	Sulphuric acid
hv	The energy of a photon
$KMnO_4$	Potassium permanganate
MG	Malachite Green
NHE	Normal Hydrogen Electrode
N_2	Nitrogen
Ni	Nickel
NO_2	Nitrogen dioxide
NO_3	Nitrate
O_2	Oxygen
$O_2 \bullet^-$	Superoxide radical

•OH	Hydroxyl radicals
PANI	Polyaniline
pH	Potential of Hydrogen
PL	Photoluminescence spectroscopy
ppm	Parts per Millions
rGO	Reduced Graphene Oxide
SnO_2	Tin (IV) oxide
SSM	Stainless steel mesh
TEOA	Triethanolamine
TiO_2	Titanium oxide
TOC	Total organic carbon
UV	Ultraviolet visible
(UV–Vis DRS)	Ultraviolet–visible diffuse reflectance spectroscopy
VB	Valence band
XRD	X-ray diffraction
ZnO	Zinc Oxide

CHAPTER 1

INTRODUCTION

1.1 Research Background

Water is a necessary resource for all living creature. The technology development and innovation has improved human access to safe and adequate water supply system. Yet, there are rising concern of water scarcity due to industrialisation, urbanisation, irrigating agriculture and population growth that affect the cleanliness and the demand of water (Hannah et al., 2022). History of water pollution has three phases: the chronic organic pollution as lack of faecal waste treatment, diffuse pollution from agriculture, mining, forestry, industrial sector and the emerging contaminant that may invisible from industrial, medical and pharmaceutical. Hence, authority must constantly monitor and create awareness among the citizen to avoid water pollution. Moreover, change of land-use and global climate change bothered the experts' perturbation toward water crisis (Levia et al., 2020).

Nowadays, human have significant favour to keep up with trending outfit had contributed to the blooming of fashion industry. From the aspect of environmentalist, textile dye can triggered environmental hazard and risk of reducing water quality (Uddin, 2021). As textile dye significantly pollute water resource that consequently increase biochemical oxygen demand, inhibit photosynthesis of aquatic plant and poisonous aquatic living. Although dyes were used since ancient times, reports showed synthetic dye such as azo dye posses mutagenic, toxic, and carcinogenic properties which is detrimental to human health and aquatic living (Shindhal et al., 2021).

Malachite green is an azo dye that has multifunction such as textile dye, counter stain for histology, antifungal agent, antibacterial agent in agriculture and etc. However, Malachite Green was reported as an toxic substances that carcinogenesis, mutagenesis, teratogenicity and respiratory toxicity (Srivastava, Sinha, & Roy 2004). Hence, removal of residue Malachite Green dye after industrial process was necessary for the sack of environmental and living organisms.

Remediation of textile dye was an important affair for environment sustainability to avoid inevitable adverse impact to the Mother Nature. There are numerous wastewater treatment such as membrane filtration, adsorption, advanced oxidation processes (AOP), bio-microbial degradation, bio-electrochemical system. Each treatment method offer advantages and limitation in dye removal process (Shindhal et al., 2021).

In recent years, photocatalysts gain massive attention from researchers as a promising resolution to air, water contamination or soil pollution due to its environment friendly nature (Asmatulu & Khan 2019; Zhou et al. 2014). Researcher also successfully developed photocatalyst to achieve photocatalytic production of hydrogen, H_2 (Xiang, Yu, & Jaroniec 2012). Scientists were urged to develops earth- abundant, low-cost, stable photocatalyst, with high efficiency for utilization in a broad solar-light spectrum to generate hydrogen. Moreover, photocatalyst can be practically used to degrade dyes which was largely used in paper, fashion and food industry.

1.2 Problem Statement

Despite adsorption method that transfer pollutant from mobile state to coagulated state. Dye photodegradation helps to break down pollutant and deteriorate it to inoculant particle using photocatalyst. Tremendous amount of research on water pollutant photodegradation via photocatalysis method have been review and published as literature (Gamage & Zhang 2010). Concerning the vital role of water to global life, purification of domestic and industrial wastewater for reusable purpose was desirable for sustainable development (Ayodhya & Veerabhadram 2018).

Photocatalyst functionality was greatly depend on the material properties to achieve excite state via photon absorption followed by photoreaction process. Hence the material selection for photocatalyst was crucial. A highly functional photocatalyst material has appropriately narrow optical band gap as it required to be photosensitive and capable to capture wider range of light spectrum. From previous research, band gap of a photocatalyst can be adjusted with construction of heterostructure photocatalyst or manipulate the synthesis method and condition to alter photocatalyst morphology (Ayodhya & Veerabhadram 2018; Shindhal et al. 2021). Moreover, adequate absorptivity between photocatalyst and targeted pollutant will influence the photodegradation efficiency. However, the challenge in photocatalysts construction was fulfil the expectation of high performance efficient at cheaper cost with environmentally friendly properties.

Titanium oxide (TiO_2) was one of the most common used photocatalyst material due to its natural abundance, low cost, stability, non-toxicity and high oxidative of its photogenerated holes (Anucha et al., 2022). However, TiO_2 has the wide band (3.2eV) which the light adsorption ability within ultraviolet light range only and its application as visible light activated photocatalyst was inhibited (Tsang et al., 2019).

In this research, researcher aim to develop photocatalyst with highly stable and optimal architectures to realize the wastewater photodegradation for practical applications. Iron (III) oxide, reduced graphene oxide and polyaniline were selected as raw materials among various candidate material due to its unique properties to enhance photodegradation performance. Iron (III) oxide was earth abundant compound with inexpensive cost and advantageous band gap (2.1−2.2 eV) for photon absorption in the visible light spectrum (Meng et al., 2013). While reduced graphene oxide derived from graphene oxide with fewer oxygen functional groups, exhibits high specific surface area, elasticity, and porosity. These properties enhance light absorbance, while its hydrophilic nature enables solubility in polar solvents, making it an effective scavenger agent in photocatalytic processes (Sur, 2012). Polyaniline, a notable conducting polymer, is recognized for its facile synthesis, high environmental stability, high conductivity, anti-corrosive properties, and effective protection against high temperatures and moisture, making it a versatile material for photodegradation of waste water (Boeva & Sergeyev 2014). Researcher believe the synergistic combination of these material in hybrid photocatalyst have high potential to boost the photocatalytic efficiency.

1.3 Objectives of Study

Objectives of the research are as follows:

1. To synthesis binary photocatalyst and ternary photocatalyst using reduced graphene oxide, iron (III) oxide and polyaniline using in situ oxidation method

2. To characterise the prepared binary photocatalyst and ternary photocatalyst using different characterisation instruments.

3. To examine the efficiency of the binary and ternary photocatalysts for photodegradation of dye in water.

1.4 Scope of study

The research scopes are in accordance with the research objectives of this study.

Research Scope for Objective 1: Reduced graphene oxide and iron (III) oxide were ultrasonicated followed by in situ oxidation method to prepare binary photocatalyst.

Three weight ratio of binary photocatalyst, FG (1:1), FG (1:2) and FG (2:1) were prepared. On the other hand, reduced graphene oxide, iron (III) oxide and polyaniline were used to prepare ternary photocatalyst using ultrasonicated in situ oxidation method. Three weight ratio of binary photocatalyst, PFG (1:2), PFG (1:2) and PFG (2:1) were prepared. The hybridization of photocatalyst with reduced graphene oxide, polyaniline and iron (III) oxide was aimed to produce a composite with suitable and well bonded structure in order the composite to function as high efficient photocatalyst. Moreover, researcher will optimize the material molar ratio to produce hybrid ternary photocatalyst with optimum photocatalytic effect.

Research scope for Objective 2: Through incorporation of polyaniline and iron (III) oxide with reduced graphene oxide, the physiochemical properties of the composite was expected to be different from the bare reduced graphene oxide nanocomposite especially the optoelectronic and charge transfer properties of the composite. The reduced graphene oxide, polyaniline and iron (III) oxide synthesized were characterized for its morphological, microstructures, arrangement and spacing of atoms, optical absorption spectrum and chemical functional groups. Instrument used included field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD), Ultraviolet–Visible Diffuse Reflectance Spectroscopy (UV–Vis DRS) and Fourier-transform infrared spectroscopy (FTIR). Photoluminescence spectroscopy (PL) were used to analyse the functionality and density of the charge while examine the recombination of the electron-hole pair and the lifetime of the charge of the composite. Brunauer-Emmett-Teller (BET) surface area analysis was used to examine the surface area of photocatalyst composites.

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Research scope for Objective 3: The feasibility of binary and ternary photocatalyst to perform photocatalytic process was examined with dye photodegradation using a particulate suspension system. In this case, the photocatalytic activity for dye photodegradation was performed under Ultraviolet (UV) lamp irradiation.

1.5 Significant of Study

In this research, degradation of textile dye was operated with photocatalyst composite with light irradiation as energy source. This is because sunlight energy was an abundant and alternative renewable source that contributed less environmental footprint compare to fossil fuels. The radiant energy from Sun travel 150 million kilometres to earth planet with speed of light, 3.0 x 10^8 km s^{-1}. With some portion of light reflected and absorbed by the ozone layer, the energy reaches our earth surface was less than 1%. Amuse to discover the energy provide by sun is still in ample amount. The theoretical calculation energy harvested from one hour sunlight was equivalent to one year energy usage by American (Skipka & Theodore 2014). Solar energy has advantageous as a clean energy source with potential to become scalable without huge environmental impact as the building water turbine dam that destroy ecosystem and natural habitat. In short, with proper enhancement of technology, sun can provide human abundance energy for a sustainable living style.

Human often utilise various technology device to capture the solar energy and converted to light, heat, electricity or ventilation system in home or industry building.

These technology devices included photovoltaic system's solar panels, solar walls, solar heater, solar paving, solar transport vehicle and etc. It is a good ideal to direct utilise energy from sun to help degrade the toxic textile dye. Well-designed heterostructure semiconductor with proper narrow band gap can harvest much natural sunlight spectrum under low charge recombination rate (H. Wang et al., 2014).

In 1970, Fujishima and Honda constructed a commercialise semiconductor TiO_2 electrode which can be activated by UV light to allow water cleavage or water splitting occur thus induce chemical conversion reaction. In other words, water can be decomposed to hydrogen and oxygen with UV light and TiO_2 semiconductor without external voltage. This semiconductor electrode's electrons excite and holes formed when adequate light strikes TiO_2 electrode connected with platinum black electrode. Holes formed react with water molecule and undergoes oxidation. While, the electrons reduce the hydrogen ions to form hydrogen (Dahal, 2016; Fujishima & Honda, 1972).

$$TiO_2 + 2hv \rightarrow 2h^+ + 2e^- \quad (1.1)$$

$$H_2O + 2h^+ \rightarrow ½\, O_2 + 2H^+ \quad (1.2)$$

$$2H^+ + 2e^- \rightarrow H_2 \quad (1.3)$$

Overall reaction from equation 1.2 and 1.3:

$$H_2O + 2hv \rightarrow ½\, O_2 + H_2 \quad (1.4)$$

While a number of light driven photocatalysts have been proposed for these purposes. These photocatalysts face challenge from low quantum efficiency and poor stability, a satisfactory material has yet to be devised. Therefore, this research aims to develop and study the fundamental effect of graphene oxide-based polymer as a cheap and abundant photocatalyst. The optimization of the heterojunction, binary and ternary photocatalyst were aimed to display optimal conduction and valence band edge alignment and corrosion-resistant with low cost and high photocatalytic performance. Then, a heterojunction structure of composite will be systematically investigated to prepare photocatalyst heterojunctions for dye wastewater degradation under sunlight. To improve photocatalytic process to its maximum value, researcher need to understand the photocatalytic principle and mechanism. Subsequently customize a photocatalyst enable the photocatalyst to poses suitable electron transition level for the targeted application.