A FACILE SURFACTANT-ASSISTED SYNTHESIS OF GRAPHENE OXIDE/ZINC OXIDE CATALYST FOR THE DEGRADATION OF METHYLENE BLUE DYE

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UNIVERSITI PENDIDIKAN SULTAN IDRIS

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ABSTRACT

This research aimed to study the role and the stabilization mechanism of hybrid photocatalyst comprising surfactant-exfoliated graphene oxide (sEGO) with zinc oxide (ZnO) for methylene blue (MB) removal. Graphite sheet was exfoliated to produce sEGO by employing synthesized triple chain anionic surfactant, sodium 1,4-bis(neopentyloxy)-3-(neopentylcarbonyl)-1,4-dioxobutane-2-sulfonate (TC14) and compared with commercially available sodium dodecyl sulphate (SDS). The structure, interfacial and colloidal stability of surfactant were studied using proton nuclear magnetic resonance (^{1}H NMR) spectroscopy, air-water (a/w) surface tension and zeta potential measurement. The morphology of photocatalyst were characterized using Field emission scanning electron microscopy (FESEM), Raman spectroscopy, High-resolution transmission electron microscopy (HRTEM), X-ray diffraction (XRD) and thermogravimetric analysis (TGA). The performance of photocatalyst for MB removal were measured through ultraviolet-visible (UV-vis) spectroscopy. The aggregation behaviour of sEGO in the presence of ZnO photocatalyst were analyzed using small-angle neutron scattering (SANS). Results showed that introduction of the triple-chain TC14 surfactant for sEGO with ZnO as photocatalyst demonstrated outstanding MB removal efficiency at 98.53%. SANS analysis revealed that employing TC14 surfactant enhances sEGO properties by having high surface area and rich oxygen functional group as photocatalyst compared to commercial SDS surfactant. In conclusion, the chain branching modification in surfactant chemical structure optimize sEGO properties with ZnO as efficient photocatalyst for MB removal. As implications, modification of surfactant in sEGO with ZnO as photocatalyst opens up new alternative approach for wastewater treatment.

SINTESIS MUDAH BERBANTU SURFAKTAN BAGI MANGKIN GRAFENA OKSIDA/ZINK OKSIDA BAGI PENGURAIAN PEWARNA METILENA BIRU

ABSTRAK

Kajian ini bertujuan untuk mengkaji peranan dan mekanisma penstabilan pemangkin-foto hibrid terdiri daripada surfaktan-pengelupasan grafena oksida (sEGO) dengan zink oksida (ZnO) bagi penyingkiran metilena biru (MB). Lembaran grafit telah dikelupas untuk menghasilkan sEGO dengan menggunakan surfaktan anionik berantai tiga yang disintesis iaitu 1, 4-bis (neopentiloksi) -3- (neopentiloksikarbonil) - 1, 4- dioksobutana-2-sulfonat (TC14) dan dibandingkan dengan natrium dodesil sulfat (SDS) yang terdapat secara komersial. Struktur, kestabilan antara muka dan koloid surfaktan telah dikaji menggunakan spektroskopi resonans magnet nuklear proton (^{1}H NMR), pengukuran tegangan permukaan udara-air (a/w), dan potensi zeta. Morfologi pemangkin-foto telah dicirikan menggunakan mikroskopi pengimbas pancaran medan elektron (FESEM), spektroskopi Raman dan mikroskopi penghantaran elektron resolusi tinggi (HRTEM), belauan sinar-X (XRD) dan analisis termogravimetri (TGA). Prestasi pemangkin-foto untuk penyingkiran MB telah diukur melalui spektroskopi ultralembayung-nampak (UV-vis). Kelakuan agregasi sEGO dengan kehadiran pemangkin-foto ZnO telah dianalisis menggunakan analisis serakan neutron sudut kecil (SANS). Dapatan kajian menunjukkan pengenalan surfaktan rantai tiga TC14 ke atas sEGO dengan ZnO sebagai pemangkin-foto menunjukkan kecekapan penyingkiran MB yang tertinggi pada 98.53%. Analisis SANS menunjukkan bahawa penggunaan surfaktan TC14 meningkatan sifat sEGO dengan mempunyai luas permukaan yang tinggi dan kumpulan berfungsi yang kaya dengan oksigen sebagai pemangkin-foto berbanding dengan surfaktan komersial SDS. Kesimpulannya, pengubahsuaian rantai bercabang dalam struktur kimia surfaktan mengoptimumkan sifat sEGO dengan ZnO sebagai pemangkin-foto yang cekap untuk penyingkiran MB. Sebagai implikasinya, pengubahsuaian surfaktan dalam sEGO dengan ZnO sebagai pemangkin-foto membuka pendekatan alternatif baru untuk rawatan air sisa.

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AOPs	Advanced oxidation processes
A_{cmc}	Limiting area per molecule at cmc
a/w	Air-water
Bi_2WO_6	Russellite
CB	Conduction band
Cd^{2+}	Cadmium ion
CdS	Cadmium sulfide
$cm^2V^{-1}S^{-1}$	Square centimeters per volt per siemens
cmc	Critical micelle concentration
CO_2	Carbon dioxide
Co_3O_4	Cobalt(II,III) oxide
CVD	Chemical vapour deposition
DLS	Dynamic light scattering
Eg	Band gap energy
eV	Electronvolt
FESEM	Field emission scanning electron microscopy
GO	Graphene oxide
GPa	Gigapascals
H_2O	Water

H_2SO_4	Sulfuric acid
HRTEM	High-resolution transmission electron microscopy
I(Q)	Scattering intensity
I_D/I_G	Ratio of defect and graphitic band intensity
K	Kelvin
$KMnO_4$	Potassium permanganate
MB	Methylene blue
meV	Millielectronvolt
MO dye	Methylene orange dye
P(Q)	Form factor
pH	Potential of hydrogen
rGO	Reduced graphene oxide
SANS	Small-angle neutron scattering
SDS	Sodium dodecylsulfate
SLD	Scattering-length density
SnO_2	Tin oxide
$SrTiO_3$	Strontium titanate
sEGO	Surfactant-assisted exfoliation graphene oxide
S(Q)	Structure factor
TC14	Sodium 1,4-bis(neopentyloxy)-3-(neopentyloxycarbonyl)-1,4-dioxopentane-2-sulfonate
TGA	Thermogravimetric analysis
TiO_2	Titanium dioxide
TMS	Tetramethylsiloxane

UV	Ultraviolet
$V(h)$	Potential energy
VB	Valence band
WO_3	Tungsten oxide
XRD	X-ray diffraction
ZnO	Zinc oxide
ZrO_2	Zirconium dioxide
Γ	Adsorption isotherm
γ_{cmc}	Surface tension at cmc
ζ-potential	Zeta potential
^1H NMR	Proton nuclear magnetic resonance

CHAPTER 1

INTRODUCTION

1.1 Research Background

Water, as a fundamental necessity, profoundly influences our daily lives. However, the escalating challenges of water pollution, rapid urbanization, and a burgeoning global population have thrust humanity perilously close to a severe water resource shortage. A stark revelation from the United Nations World Water Development Report (2017) underscores the alarming reality that over 80% of wastewater globally, and surging to over 95% in certain least developed nations, is released into the environment without undergoing any treatment. This dire situation underscores the crucial role of wastewater management, as highlighted by the report, not only in mitigating pollution but also in generating social, environmental, and financial benefits essential for sustainable development. The report emphasizes the urgency of adopting responsible wastewater management practices to address these multifaceted challenges. It is within this context that the work presented here gains paramount significance, contributing to the collective effort required to safeguard water resources and foster a sustainable future. The latest update from the United Nations on wastewater treatment outlines ambitious goals aimed at enhancing water quality globally. The strategies involve reducing pollution, eliminating improper dumping practices, and restricting the discharge of hazardous materials and chemicals into water bodies. The overarching objective is to diminish untreated pollution significantly. A key target set by the United Nations is that, by the year 2030, the entire world will adopt comprehensive measures for recycling and reusing water resources (Alabaster, Johnston, Thevenon, & Shantz, 2021). In the context of these global initiatives, the work conducted here gains heightened importance. The research and advancements in water treatment technologies discussed in this context contribute directly to the collective effort to achieve these worldwide goals, aligning with the United Nations' vision for a sustainable and responsibly managed water future.

The escalating contamination of water systems, encompassing streams, rivers, and lakes, has emerged as a pressing issue in contemporary times. This predicament is largely attributed to the unregulated discharge of industrial pollutants without adequate treatment. Notably, textile industries significantly contribute to the release of wastewater laden with heavy metals and non-biodegradable organic dyes (Saravanan, Gracia, & Stephen, 2017). Among these pollutants, methylene blue (MB) dye, an extensively employed aromatic cationic dye in paper, agrochemical, and textile industries, stands out as a pervasive environmental contaminant (González, Villanueva, Piehl, & Copello, 2015). Its introduction into water bodies imparts undesirable color, rendering the water unsuitable for various purposes, including drinking. Given the persistence of these dyes and their adverse effects on living organisms through prolonged exposure, concerted efforts are imperative to preserve the cleanliness of water sources. Consequently, the continuous removal of such pollutants from water assumes paramount importance in ensuring a sustainable and healthy environment.

Various techniques, including coagulation, biodegradation, adsorption, and membrane filtering, have been employed for the remediation of colored wastewater. However, these conventional approaches have demonstrated limitations in completely degrading contaminants in polluted water. Membrane technologies, while effective, are expensive and may lead to the generation of secondary pollutants, contrasting with adsorption and coagulation, which primarily transfer pollutants to different phases (Anjum, Miandad, Waqas, Gehany, & Barakat, 2019; Fei et al., 2018; Katheresan, Kansedo, & Lau, 2018). There is an urgent need for the development of environmentally friendly methods capable of efficiently breaking down dye pollution, considering the drawbacks associated with current approaches.

The rising demand for efficient water treatment has led to significant attention on advanced oxidation processes (AOPs), particularly those involving the generation of reactive oxygen radicals capable of reacting with various pollutants. Among these AOPs, photocatalysis stands out as a promising approach, utilizing light and semiconductors (Banerjee, Benjwal, Singh, & Kar, 2018; Khataee & Kasiri, 2010; Martins et al., 2018). Photocatalysis offers high degradation efficiency, effectively breaking down diverse organic compounds with low energy consumption. However, challenges such as interfacial charge transfers, inhibition of charge carrier recombination, catalyst preparation techniques, and reactor design can impact its efficacy (Iervolino, Zammit, Vaiano, & Rizzo, 2020). Addressing these challenges becomes crucial in advancing photocatalytic technologies for tackling the persistent issue of dye pollution in water bodies.

Semiconductor materials are crucial for activating photon excitation in photocatalysis processes, with zinc oxide (ZnO), titanium dioxide (TiO_2), cadmium sulfide (CdS), tungsten oxide (WO_3), tin oxide (SnO_2), strontium titanate ($SrTiO_3$), russellite (Bi_2WO_6), and zirconium dioxide (ZrO_2) being frequently employed in such applications (Gaya, 2014). Notably, according to reports by Anjum et al. (2019) and Adnan et al. (2016), ZnO stands out as the ideal semiconductor photocatalyst due to its exceptional attributes. ZnO exhibits high electron mobility, solid thermal and mechanical stability, elevated quantum efficiency, formidable resistance to degradation, potent oxidizing ability, and an expansive surface area, making it highly suitable for efficient photocatalytic processes. This recognition emphasizes ZnO's pivotal role in harnessing light energy for effective pollutant degradation. The efficiency of the ZnO photocatalyst faces challenges due to the tendency of ZnO particles to aggregate, resulting in a decrease in the accessibility of active surface sites (Azmina et al., 2017). Additionally, Sun et al. (2018) highlight that ZnO's broad bandgap energy (3.37 eV) promotes rapid recombination of electron-hole pairs, potentially diminishing the effectiveness of its photocatalytic activity. These issues underscore the importance of addressing aggregation tendencies and bandgap energy limitations to enhance the overall performance of ZnO in photocatalysis. ZnO is frequently susceptible to hybridization with other semiconductors, metals, non-metals, or carbon-based materials to get over these restrictions and improve its photocatalytic capabilities. Graphene oxide (GO) is a more modern and promising option. The high rate of electron-hole pair recombination within ZnO is a problem that can be mitigated by combining ZnO with similar materials (Y. Sun, Zhang, Li, Liu, & Wang, 2023). By exploring these synergistic approaches, we can unlock the full potential of ZnO-based photocatalysts, paving the way for novel and improved solutions in photocatalytic applications.

Numerous reports have highlighted the significant enhancement of photocatalytic efficiency through the incorporation of graphene oxide (GO), owing to the rapid electron transfer facilitated by the sp^2 hybridization of GO carbon atoms (N. Sun et al., 2018). Currently, the widely favored method for synthesizing GO involves the Hummers' approach, which yields a substantial amount of product. However, this method comes with drawbacks, particularly its reliance on environmentally unfriendly chemicals such as strong oxidizing agents like $H_2SO_4/KMnO_4$ and excessive use of organic solvents like dimethylformamide or tetrahydrofuran (Moosa & Abed, 2021). In response to these concerns, researchers have explored alternative environmentally friendly approaches, one of which involves an electrochemical method assisted by electrical power to generate GO. Traditionally, GO is produced through the oxidation and exfoliation of graphite electrodes in the presence of electrolytes, often utilizing inorganic salts and aqueous acids (Z. Xue, Zhao, Zhao, Li, & Gao, 2016). However, this method necessitates neutralization of the resulting GO suspension for further applications.

In the context of graphene oxide (GO) synthesis, an intriguing alternative to the conventional use of salts and acids involves the utilization of surfactant solutions in the exfoliation electrolyte. This surfactant-assisted strategy not only simplifies and enhances the safety of the process compared to the Hummers' method but also imparts additional stability to the resulting GO suspension (Pendolino & Armata, 2017). In the electrochemical exfoliation method, the inclusion of surfactants plays a pivotal role in facilitating the intercalation and exfoliation processes of graphite, contributing to the formation of oxidized graphene oxide (GO) (Narayan, Lim, Jeon, Li, & Kim, 2017). The surfactant-facilitated exfoliation of graphite represents an appealing and promising approach for the efficient production of GO (Hu, Su, Xie, Sun, & Kou, 2019).

1.2 Problem Statement

Zinc oxide (ZnO), often utilized as a semiconductor material in photocatalytic applications, confronts challenges associated with its separation from treated water and a propensity to aggregate, resulting in a reduction of its photocatalytic efficiency (Mousavi, Davar, & Loghman-Estarki, 2016). Moreover, the use of ZnO in powder form proves ineffective, yielding a milky solution that is easily dispersed (Katheresan et al., 2018). To mitigate these challenges, carbonaceous materials such as graphene oxide (GO) can be employed to enhance photocatalytic performance further. Owing to their exceptional strength, high conductivity, and extensive surface area, these materials hold the potential to stabilize electron/hole separation by acting as electron acceptors and carriers (Mallakpour & Rashidimoghadam, 2018; Saleh, 2013). According to Kang et al. (2016), Hummer's method, is frequently used for high-quality GO production. However, this approach involves the use of hazardous chemicals and potent acids that can severely pollute the environment, demand extended production times, and leave metal ion impurities on GO sheets (Pei, Wei, Huang, Cheng, & Ren, 2018; Suriani et al., 2018). In response to these concerns, the electrochemical exfoliation approach was chosen for GO synthesis due to its ease of use, cost-effectiveness, environmental friendliness, and the ability to operate at room temperature and pressure (Jaiswal, Modak, & Devi, 2024). In the context of the photocatalytic degradation of methylene blue, this dye presents a distinct set of challenges. Methylene blue is commonly used in various industries and its removal from wastewater is crucial due to its harmful effects on the environment and human health (Oladoye, Ajiboye, Omotola, & Oyewola, 2022). The primary issue lies in its resistance to degradation under conventional treatment methods. Therefore, an efficient and sustainable approach, such as advanced photocatalysis, becomes imperative for its successful removal. However, the combination of graphene oxide and zinc oxide, while promising for enhanced photocatalysis, also encounters certain challenges. The tendency of ZnO to aggregate and the difficulty in its separation from the treated water can hinder the overall efficiency of the photocatalytic process (Raizada, Sudhaik, & Singh, 2019). Additionally, achieving proper dispersion and interaction between GO and ZnO is crucial for optimizing their synergistic effects in promoting electron/hole separation and facilitating the photocatalytic degradation of pollutants. Since the surfactant solely serves as a stabilizing agent, recent literature has mostly concentrated on the efficiency and optimization of exfoliation (Md Disa et al., 2015). The exploration and refinement of surfactant chemical structures play a pivotal role in achieving the desired outcomes, particularly in the context of wastewater treatment.

1.3 Aim and Research Objectives

The aims were to systematically study the role and the stabilization mechanism of graphene-compatible surfactants for the production of graphene oxide-zinc oxide nanocomposite and their applications for the degradation of methylene blue.

The research objectives of this study are:

- To synthesize and characterize graphene-compatible surfactant.
- To produce surfactant-assisted exfoliation graphene oxide (sEGO) through the electrochemical exfoliation method.
- To fabricate sEGO-ZnO photocatalyst for the degradation of methylene blue dye.

1.4 Scope of Study

The primary objective of these research initiatives is to bridge existing knowledge gaps by leveraging hyperbranched surfactant structures to optimize the synthesis of graphene oxide, subsequently applied in the realm of photocatalytic wastewater treatment. Surfactants, specifically a commercial single chain (SDS) and a custom-made triple-chain surfactant (TC14), were instrumental in facilitating the electrochemical process employed for graphene oxide production.

To elucidate the chemical structures of the surfactants, proton nuclear magnetic resonance (^{1}H NMR) spectroscopy was employed. The surface tension of surfactant solutions in air-water (a/w) systems was measured using a Willhelmy tensiometer, while zeta (ζ) potential analysis was concurrently utilized to assess the surface charge of surfactant-stabilized exfoliated graphene oxide (sEGO). Moreover, small-angle neutron scattering (SANS) was applied to investigate the shapes and sizes of the self-assembled materials.

A comprehensive characterization of the structural and morphological features of both sEGO and the sEGO-ZnO catalysts was achieved through the synergistic application of Field Emission Scanning Electron Microscopy (FESEM), High-Resolution Transmission Electron Microscopy (HRTEM), Raman spectroscopy and X-ray Diffraction (XRD). Photocatalytic investigations were conducted using a UV spectrophotometer, and the thermal characteristics of the catalysts were examined through Thermogravimetric Analysis (TGA).

This multi-faceted approach integrates various analytical techniques to provide a holistic understanding of the synthesized materials, emphasizing their potential applications in the field of wastewater treatment and beyond.

1.5 Significance of Study

It has been 19 years since Andre Geim and Konstantin Novoselov first discovered graphene. There is a vast of research on the photocatalytic use of GO with different production and modification techniques. However, the specialized production of GO using one-pot electrochemical exfoliation using modified surfactant for water treatment receives less attention. As a result, the primary purpose of this work is to investigate how the introduction of a terminal methyl group in the surfactant chain can improve sEGO characteristics with ZnO for photocatalytic applications. The next step is to look into how the generated sEGO might be used to improve photocatalytic studies of methylene blue removal in an aqueous solution. Moreover, contemporary research has predominantly concentrated on optimizing the exfoliation process, emphasizing efficacy, rather than delving into the fundamentals of contaminant and dye removal. In this context, the surfactant primarily serves as a stabilizing component (Sham & Notley, 2018). The ultimate objective is to develop surfactants that support effective exfoliation and dye removal simultaneously.

A study was done on the functions of sEGO as an efficient catalyst for the removal of methylene blue from an aqueous solution. Various initial methylene blue concentrations, pH levels, and dosages were used in the photocatalytic investigations. The absorbance value information was gathered using UV-visible spectroscopy. To evaluate the removal behavior of MB by sEGO-metal oxide, the experiment data from batch photocatalytic investigations was examined and interpreted for photodegradation performance. The findings demonstrate new potential for employing sEGO as a catalyst in direct (in situ) applications and lay the groundwork for the creation of future surfactants for water treatment systems based on carbon nanomaterials.