INCORPORATION OF Fe AND gC_3N_4 INTO MESOPOROUS TITANIA NANOPARTICLES FOR PHOTODEGRADATION OF DIBENZOTHIOPHENE

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UNIVERSITI PENDIDIKAN SULTAN IDRIS

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ABSTRACT

This study aimed to incorporate Fe and gC_3N_4 into Mesoporous Titania Nanoparticles (MTN) and investigate photodegradation of Dibenzothiophene (DBT). MTN with various aging time (x = 2, 4, 6 and 8 days) were synthesized via sol-gel method before being incorporated with 5wt% Fe (5Fe/MTN-x) and 5wt% gC_3N_4 (5FeMTN-x/5gC_3N_4) using wet-impregnation method. The photocatalysts were characterized using X-ray Diffraction (XRD), Field-Emission Scanning Electron Microscopy (FESEM) with EDX analysis, UV-Visible Diffuse Reflectance Spectroscopy (UV-DRS), Fourier-Transform Infrared Spectroscopy (FTIR), Surface Area Analysis (BET), Photoluminescence Spectroscopy (PL) and Raman Spectroscopy. Photocatalytic performance was evaluated through DBT photodegradation under ultraviolet (UV) light irradiation for 180 min. Parameters such as H_2O_2 volume, DBT concentration and photocatalyst dose were manipulated with prolonging the reaction time to 240 min. The results of XRD showed the formation of MTN with the presence of common peaks at 2θ: 25.34^o - 75.00^o corresponded to TiO_2 anatase phase. FTIR and BET analyses also confirmed the formation of MTN and successful incorporation of Fe and gC_3N_4 into the hybrid catalysts. UV-DRS and PL results showed the extending of light absorption towards visible light region and lower rate of electron-hole recombination was reached by the addition of Fe and gC_3N_4. The results displayed 5FeMTN-6/5gC_3N_4 owned the highest DBT photodegradation with 38.2% compared to the other photocatalysts. At the optimized conditions, 5FeMTN-6/5gC_3N_4 reached 57.0% at 40 mg/L DBT concentration with 1 g/L photocatalyst dose and 0.01 mL H_2O_2. The presence of Fe and gC_3N_4 enhanced the photocatalytic performance by forming a heterojunction that promoted the generation and inhibit the electron-hole recombination. In conclusion, Fe and gC_3N_4 were successfully incorporate into MTN and reached 57.0% DBT photodegradation at optimized conditions. The implication of this study is this photocatalyst has good potential to be further investigated as a low-cost alternative for DBT removal in fuels.

PENGGABUNGAN Fe DAN gC_3N_4 KE DALAM NANOZARAH TITANIA MESOFORUS UNTUK FOTODEGRADASI DIBENZOTIOFENA

ABSTRAK

Kajian ini bertujuan untuk menggabungkan Fe dan gC_3N_4 ke dalam Nanozarah Titania Mesoforus (MTN) dan mengkaji fotodegradasi Dibenzotiofena (DBT). MTN dengan masa penuaan berbeza (x = 2, 4, 6 dan 8 hari) disintesis melalui kaedah sol-gel sebelum digabungkan bersama 5wt% Fe (5Fe/MTN-x) dan 5wt% gC_3N_4 (5FeMTN-x/5gC_3N_4) menggunakan kaedah penjejalan basah. Sampel fotopemangkin dicirikan menggunakan analisis Pembelauan Sinar-X (XRD), Mikroskopi Pengimbasan Elektron Pancaran Medan (FESEM) dengan analisis EDX, Spektroskopi Resapan Pantulan Ultralembayung (UV-DRS), Spektroskopi Inframerah Fourier Transformasi (FTIR), Analisis Luas Permukaan (BET), Spektroskopi Fotoluminasi (PL) dan Spektroskopi Raman. Prestasi fotopemangkin dinilai melalui fotodegradasi DBT di bawah sinaran ultralembayung (UV) selama 180 min. Parameter seperti isipadu H_2O_2, kepekatan DBT dan dos fotopemangkin telah dimanipulasi dengan memanjangkan masa tindak balas kepada 240 min. Dapatan XRD menunjukkan pembentukan MTN dengan kehadiran puncak biasa pada 2θ: 25.34^o - 75.00^o sepadan dengan fasa *anatase* TiO_2. Analisis FTIR dan BET juga mengesahkan pembentukan MTN dan kejayaan penggabungan Fe dan gC_3N_4 ke dalam pemangkin hibrid. Dapatan UV-DRS dan PL menunjukkan lanjutan penyerapan ke arah kawasan cahaya nampak dan kadar penggabungan semula lubang dan elektron yang lebih rendah dicapai dengan penambahan Fe dan gC_3N_4. Dapatan mempamerkan, 5FeMTN-6/5gC_3N_4 memiliki fotodegradasi DBT tertinggi sebanyak 38.2% berbanding dengan fotopemangkin yang lain. Pada keadaan optimum, 5FeMTN-6/5gC_3N_4 mencapai 57.0% pada 40 mg/L kepekatan DBT dengan 1 g/L dos fotopemangkin dan 0.01 mL H_2O_2. Kehadiran Fe dan gC_3N_4 meningkatkan prestasi fotokatalitik dengan membentuk heterosimpang yang menggalakkan penjanaan dan menghalang penggabungan semula elektron dan lubang. Kesimpulannya, Fe dan gC_3N_4 berjaya digabungkan ke dalam MTN dan mencapai 57.0% fotodegradasi DBT pada keadaan optimum. Implikasi kajian ini ialah fotopemangkin mempunyai potensi yang baik untuk dikaji dengan lebih lanjut sebagai alternatif kos rendah untuk pembuangan DBT dalam bahan api.

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LIST OF ABBREVIATIONS

ADS	Adsorptive desulfurization
AFM	Atomic Force Microscope
AO	Auto-oxidation
AOPs	Advanced Oxidation Processes
AO7	Acid Orange 7
AO10	Acid Orange 10
$BaSO_4$	Barium sulfate
BDS	Biodesulfurization
BET	Brunnauer-Emmett-Teller
BJH	Barrett-Joyner-Halenda
BPA	Bisphenol A
BT	Benzothiophene
CAL	Calibration volume
CB	Conduction band
CCDs	Charge-coupled devices
CIDs	Charge-injection devices
CM	Conventional sol-gel
2-CP	2-chlorophenol
4-CP	4-chlorophenol
CR	Congo red
Cr(VI)	Hexavalent chromium

$CSTiO_2$	Core-shell TiO_2 nanomaterials
CTAB	Hexadecyltrimethyl-ammonium bromide
CTDs	Charge transfer devices
$Cu_2(OH)_2CO_3$	Copper hydroxycarbonate
	Initial concentration
C_t	Concentration at a specific time
2D	Two-dimensional
3D	Three-dimensional
DBT	Dibenzothiophene
$DBTO_2$	DBT sulfone
2,4-D	2,4-dichlorophenoxyacetic acid
2,4 DNP	2,4 dinitrophenol
2,4-DP	2,4-dichlorophenoxypropionic acid
E	Band gap energy
e^-	Electrons
EDS	Extractive desulfurization
EDX	Energy-Dispersive X-ray
FCC	Fluidized catalytic cracking
FEG	Field emission gun
FESEM	Field-Emission Scanning Electron Microscopy
FTIR	Fourier-Transform Infrared
Fe_2O_3	Iron (III) oxide
GC	Gas Chromatography
GC-MS	Gas Chromatography-Mass Spectrometer
gC_3N_4	Graphitic carbon nitride

h^+	Holes
h	photon energy
HCl	Hydrochloric acid
HDS	Hydrodesulfurization
He-Ne	Helium-Neon
HNO_3	Nitric acid solution
$HO^\bullet_2$	Hydroperoxyl radicals
H_2O_2	Hydrogen peroxide
H_2S	Hydrogen sulphide
IR	Infrared
IWI	Incipient wet impregnation
KBr	Potassium bromide
MB	Methylene Blue
MG	Malachite Green
MO	Methyl Orange
MS	Mass spectrometry
MTN	Mesoporous TiO_2 Nanoparticles
MW	Microwave assisted sol-gel
NaOH	Sodium hydroxide
Nd:YAG	Neodymium-Yttrium Aluminium Garnet
Nd: YVO_4	Neodymium-Yttrium Ortho-Vanadate
NH_4F	Ammonium fluoride
NH_4OH	Ammonium hydroxide
4-NP	para-nitrophenol
$O_2^{\bullet -}$	Superoxide radicals

$^{\bullet}OH$	Hydroxyl radicals
ODS	Oxidative desulfurization
OV	Oxygen vacancies
Pc	Phthalocyanine
PL	Photoluminescence
PMT	Photomultiplier Tube
PODS	Photocatalytic Oxidative Desulfurization
PTFE	Polytetrafluoroethylene
PVP	Polyvinylpyrrolidone
RhB	Rhodamine B
RR	Remazol Red
RR198	Reactive Red 198
SDS	Sodium Dodecyl Sulfate
SEM	Scanning Electron Microscopy
SO_x	Sulfur oxides
SPR	Surface Plasmon Resonance
STM	Scanning Tunneling Microscope
TBOT	Titanium (IV) butoxide
TEM	Transmission Electron Microscopy
TiO_2	Titanium oxide
TIOS	Titanium (IV) oxysulfate
TNF	TiO_2 nanofiber
TNR	TiO_2 nanorod
TNT	TiO_2 nanotube
TS	Thiophene

TSD	Ti^{3+} site defects
TTIP	Titanium Tetraisopropoxide
T20	Tween 20
TX100	Triton X100
US	Ultrasound-assisted impregnation
UV	Ultraviolet
UV-DRS	UV-Visible Diffuse Reflectance Spectroscopy
XRD	X-Ray Diffraction
VB	Valence band
WO_3	Tungsten oxide
ZnO	Zinc oxide

APPENDIX LIST

CHAPTER 1

INTRODUCTION

1.1 Background Research

Desulfurization of fuel oils such as diesel, fluidized catalytic cracking (FCC) gasoline, kerosene, jet fuel and heating oil has become an important topic in oil refining industry. Sulfur compounds in fuel oils were encouraged to be removed as they create problems during refining as well as during their commercial use. During refining, these sulfur compounds can cause corrosion problems in pumping, pipeline and refining equipment and also poisoning some of catalysts used in oil processing (Bhutto et al., 2016). During the commercial use of fuel oils, sulfur compounds has released the sulfur oxides (SO_x) through combustion process which lead to environmental problems such as acid rain, global warming and atmospheric pollution. Besides, the human health was also exposed to negative impact such as respiratory illness, heart disease and asthma (Hitam, Jalil, & Triwahyono, 2018). With the heightened interest for cleaner air and increased stringent environmental regulations for sulfur concentration in fuel worldwide, the plan to achieve little-to-no sulfur fuels (S-content<10 ppm) has been implementing by many developed and developing countries in order to reduce the negative effects on environment and health (Mandizadeh, Salavati-Niasari, & Sadri, 2017; Bhutto et al., 2016). Therefore, the removal of sulfur in fuel has become the hot topic for many researchers nowadays, as intensive studies were needed to find more economical and viable deep desulfurization technologies.

Hydrodesulfurization (HDS) was the most widely used commercial method for sulfur removal in fuel oils (Bhutto et al., 2016). HDS process was indeed an efficient method in eliminating acyclic and aliphatic sulfur compounds such as thiols, sulfides and disulfides but it was difficult to reduce heterocyclic sulfur compounds such as thiophene (TS), dibenzothiophene (DBT) and its alkylated derivatives due to their steric hindrance (Hitam, Jalil, & Abdulrasheed, 2019). Besides, HDS method employs the use of hydrogen at an elevated pressure (20-100 atm of H_2) and high operating temperature (300-400 ^{o}C) which were very costly option for deep desulfurization (Zeelani, Ashrafi, Dhakad, Gupta, & Pal, 2016; Zaid, Kait, & Mutalib, 2016). Therefore, alternative ultra-deep desulfurization processes such as extractive desulfurization (EDS), adsorptive desulfurization (ADS), biodesulfurization (BDS) and oxidative desulfurization (ODS) were necessary to be developed (Mandizadeh et al., 2017).

Among those various desulfurization techniques, ODS was considered as one of the most encouraging techniques for deep desulfurization due to its mild operating conditions which can be operated under low temperature and ambient pressure, as well as its high ability to oxidize and convert refractory sulfur compound such as DBT without the hydrogen utilization (Ding et al., 2015). In fact, photocatalytic oxidative desulfurization (PODS) has gained attention as the promising technique to improve the performance of ODS, since photocatalysis was one of the advanced oxidation processes (AOPs) that can promote outstanding performance in degrading a wide range of toxic compounds under light irradiation. Besides, photocatalysis also provides other advantages such as easy operation, low-cost, sustainable technology, ability to perform at room temperature, environmental friendliness and capability to transform toxic contaminants into harmless products with high selectivity (Hitam et al., 2019). PODS was assumed to be a potential method to solve this problem since it promotes high catalytic activity, safety, low energy consumption and recycling ability (Zhu et al., 2014). This finding was supported by Miao et al. (2016) which stated that, PODS has been known to be a promising method as it provides a green path to photooxidize sulfur species to highly polar sulfoxides or sulfone product with the presence of ultraviolet (UV) or visible light as the energy source.

In order to improve the PODS activity, some modifications of the existing catalysts have been done by researchers. Generally, the photocatalytic efficiency can be improved by several techniques such as synthesize of high surface area materials, the use of support material, coupling with other semiconductors, formation of a defect structure to improve charge-carrier separation and coupling with organic or inorganic materials (Hitam et al., 2019).

Over the last few decades, various photocatalysts have been developed for PODS and semiconductor-based catalysts such as zinc oxide (ZnO), titanium oxide (TiO_2), tungsten oxide (WO_3), etc were known to exhibit excellent photocatalytic performance (Kang et al., 2019). The presence of photocatalyst in PODS was crucial as it promotes the oxidation of sulfur-containing compounds by enhancing the rate of active species formation and improving the charge transfer. From previous studies, the instantaneous increased of sulfur removal depends on the increasing amount of photocatalyst (Aazam, 2014; Abdelaal & Mohamed, 2014). However, the decreasing in sulfur removal was occurred as the further addition of photocatalyst was beyond the optimum value. It was due to the excess amount of catalyst tends to inhibit the light source and decrease photoelectron productivity which results in reducing the photocatalytic oxidation efficiency (Li et al., 2016). In this perspective, TiO_2 as semiconductor photocatalyst has received considerable attention due to its commercial availability, high chemical stability and excellent catalytic properties (Petronella et al., 2017). This finding was supported by Hosseini and Faghihian (2019) which stated that, TiO_2 was one of various semiconductors that has been widely utilized in photodegradation experiments due to its elevated photocatalytic activity, photochemical stability, affordability and safe to use.

1.2 Problem Statement

TiO_2 has been widely used in the PODS of DBT in fuel due to its promising photoactivity, non-toxicity, chemical stability and low cost (Hitam et al., 2019). However, TiO_2 has several drawbacks that limit its practical application such as has narrow spectrum utilization due to its wide band gap (3.2 eV) and rapid recombination of electron-hole pairs (Jaafar, Jalil, & Triwahyono, 2017). Modification of TiO_2 into a mesoporous structure was reported successfully increased the stability and photoactivity of the photocatalyst (Zhang et al., 2020). Besides that, the modification of TiO_2 with transition metal and polymeric carbon material can also enhance the photoactivity of the photocatalyst (Sadanandam, Zhang, & Scurrell, 2018; Zaid et al., 2016). Among those various metallic dopants, Fe was considered to be the promising candidate for this research due to the ionic radius of Fe^{3+} (0.69 Å) was nearly similar to Ti^{4+} (0.745 Å), which in turn can help the Fe^{3+} ions to be easily incorporated into TiO_2 crystal lattice. As a result, Fe^{3+} will restrict the recombination of electron-hole pairs by performing as a charge carrier trap, which can significantly enhance the photoactivity (Marami, Farahmandjou, & Khoshnevisan, 2018). In recent years, gC_3N_4 as a well-known polymer has got much attention from researchers due to its advantages such as low cost, electronic characteristics, high chemical and thermal stability, and also an efficient non-metallic photocatalyst (Gahlot, Dappozze, Mishra, & Guillard, 2021; Abedini, Allahyari, & Rahemi, 2021). The incorporation of both Fe and gC_3N_4 can improve the separation of photogenerated electron-hole pairs as it can form heterojunction known as Z-scheme mechanism that allow the reduction and oxidation reactions occurred from different sites of photocatalyst (Zhu, Murugananthan, Gu, & Zhang , 2018). Nevertheless, there was no study has been done to investigate the potential of mesoporous TiO_2 with incorporation of Fe and gC_3N_4 in the degradation of DBT under UV light yet.

1.3 Research aim

The aim of this research was to investigate the photocatalytic efficiency of mesoporous TiO_2 nanoparticles (MTN) with addition of Fe and gC_3N_4 for the photodegradation of DBT in model fuel under UV light irradiation.

1.4 Research Objectives

The objectives of this research are:

1) To synthesize MTN via sol-gel method and modification with Fe and gC_3N_4 via wet-impregnation method.
2) To study the physicochemical properties of prepared photocatalysts.
3) To elucidate the photocatalytic activities of prepared photocatalysts in photodegradation of DBT under UV light.

1.5 Scope of study

In this research, MTN was synthesized via sol-gel method and modified with Fe and gC_3N_4 as a photocatalyst for the photodegradation of DBT in model oil under UV light irradiation. Different aging time (x = 2, 4, 6 and 8 days) were used to prepare the bare MTN-x. Then, 5wt% of Fe was added into MTN-x to form 5Fe/MTN-x before being modified with 5wt% of gC_3N_4 dopant by wet-impregnation method and denoted as 5FeMTN-x/5gC_3N_4. Then, the physicochemical properties of prepared photocatalysts were characterized using X-ray diffraction (XRD), Surface area analysis (BET), UV-Visible diffuse reflectance spectroscopy (UV-DRS), Field-Emission scanning electron microscopy (FESEM), Fourier-transform infrared spectroscopy (FTIR), Photoluminescence (PL) and Raman spectroscopy. The photocatalytic activity of 5FeMTN-x/5gC_3N_4 was compared with undoped MTN-x and also 5Fe/MTN-x in order to determine the effectiveness of the combination of Fe and gC_3N_4 into MTN for photodegradation of DBT in model oil under UV light. After photocatalytic reaction, the degraded DBT solution was characterized by GC-MS to determine the degradation product.