

SYNTHESIS OF CALCIUM FERRITE
PHOTOCATALYST FOR THE COD
PHOTODEGRADATION OF PALM OIL MILL
EFFLUENT UNDER VISIBLE LIGHT

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DOCTOR OF PHILOSOPHY

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We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.

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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Malaysia telah lama bergelut dengan isu pencemaran efluen kilang minyak sawit (POME) yang terbukti menjadi isu alam sekitar yang serius. Walaupun beberapa kajian untuk mengatasi masalah ini dengan menggunakan fotokatalis ZnO dan TiO₂ di bawah sinaran ultralembayung telah dilaksanakan, kajian tersebut masih ada ruang untuk penambahbaikan. Efisiensi cahaya rendah dan kecekapan photodegradation sederhana yang dihasilkan oleh dua sistem ini menonjolkan keperluan untuk fotokatalis yang diaktifkan oleh cahaya nampak yang dapat dilihat sebagai penyelesaian jangka panjang kepada masalah pencemaran alam yang disebabkan oleh POME. Tesis ini menghuraikan penggunaan CaFe₂O₄ sebagai fotokatalis yang diaktifkan oleh cahaya nampak untuk menangani masalah tersebut. Dua kaedah sintesis (pembakaran automatik dan permendakan bersama) dan dua suhu kalsinasi (550 °C dan 700 °C) digunakan untuk menghasilkan sejumlah empat pemangkin CaFe₂O₄ yang digelar AC550, AC700, CP550 dan CP700. CP550 mempamerkan penyingkiran COD terbesar sebanyak 69% pada kepekatan pemangkin 0.75 g/L selepas 8 jam irradiasi. Reaksi ini mematuhi kinetik urutan pertama dengan pemalar kadar $2.7 \times 10^{-5} \text{ min}^{-1}$. Analisis BET menunjukkan bahawa CP550 mempunyai isipadu S_{BET} tertinggi (27.28 m²/g) dan isipadu liang tertinggi (0.077 cm³/g) antara semua fotokatalis yang diuji yang menurun secara mendadak bila suhu kalsinasi ditingkatkan untuk CP700 menyebabkan susutan S_{BET} kepada 9.73 m²/g dan isipadu liang 0.025 cm³/g disebabkan oleh penyepuhlandapan yang menyebabkan kawasan permukaan yang licin seperti yang dapat dilihat dalam imej SEM. UV-Vis DRS menunjukkan CP550 mempunyai luang jalur tertinggi (1.52 eV) yang mungkin disebabkan oleh kehadiran CaFe₅O₇ yang merupakan struktur unik CaFe₂O₄ yang mengandungi tiga unit FeO dalam fasa stabil seperti yang disimpulkan daripada data EDX dan disahkan oleh XRD. CP550 juga memaparkan spektrum PL paling rendah antara fotokatalis yang diuji menunjukkan kadar gabungan semula lubang-elektron yang rendah. Kajian hapus sisa menggunakan IPA menyebabkan kejatuhan COD yang mendadak dari 69% kepada hanya 7% menunjukkan radikal hidroksil sebagai spesies oksidatif reaktif utama. Kajian kitar semula menunjukkan bahawa penggunaan semula pemangkin yang terpakai menyebabkan penyusutan dari segi efisiensi degradasi COD dari 69.0% hingga 65.0% dan akhirnya 61.0% selepas tiga kitaran menunjukkan kehilangan aktiviti fotokatalitik. Pencirian pemangkin yang terpakai selepas proses fotokatalisis menunjukkan bahawa kehilangan aktiviti adalah disebabkan oleh pemendapan karbon seperti yang dibuktikan oleh data FTIR dan EDX. Penyelidikan ini memberi sumbangan kepada badan pengetahuan yang menangani isu fotokatalisis yang diaktifkan oleh cahaya nampak untuk pencegahan pencemar alam sekitar yang rekalsitran seperti POME.

ABSTRACT

Malaysia has long battled with the issue of palm oil mill effluent (POME) pollution which has proven to be detrimental to the environment. Although some attempts to alleviate this problem using ZnO and TiO₂ photocatalysts under ultra-violet irradiation have been undertaken, these have shown significant room for improvement. The low light utilization as well as mediocre photodegradation efficiency produced by these two systems highlight the need for a visible light driven photocatalyst as a long term solution to the problem. This thesis explores the application of CaFe₂O₄ as a visible light driven photocatalyst towards addressing that problem. Two synthesis routes namely the auto-combustion (AC) and co-precipitation (CP) routes and two calcination temperatures (550 °C and 700 °C) were used to produce a total of four CaFe₂O₄ catalysts namely AC550, AC700, CP550 and CP700. CP550 exhibited the greatest chemical oxygen demand (COD) degradation of 69% at a 0.75 g/L catalyst loading and an oxygen flow rate of 60 ml min⁻¹ after 8 h of irradiation. The reaction adhered well to first order kinetics with a rate constant of $2.7 \times 10^{-5} \text{ min}^{-1}$. Nitrogen physisorption studies indicated CP550 had the highest Brunauer-Emmett-Teller (BET) specific surface area (27.28 m²/g) and pore volume (0.077 cm³/g) of the prepared photocatalysts which dropped precipitously for CP700 upon increasing the calcination temperature to BET specific surface area of 9.73 m²/g and pore volume of 0.025 cm³/g due to annealing which created a smoother surface area as evidenced by the scanning electron microscope (SEM) images. Ultraviolet-visible diffuse reflectance spectroscopy (UV-Vis DRS) indicated CP550 had the highest band-gap (1.52 eV) which is likely due to the presence of CaFe₅O₇ which is a unique structure of CaFe₂O₄ containing three units of FeO in a stable phase as deduced from the energy dispersive X-ray microanalysis (EDX) data and confirmed by X-ray diffraction (XRD) peaks. CP550 also displayed the lowest photoluminescence (PL) spectra of the as prepared photocatalysts indicating a low electron-hole recombination rate. A scavenging study using iso-propyl alcohol (IPA) caused a severe drop in COD degradation from 69% to just 7% indicating hydroxyl radicals as the main reactive oxidative species. The recyclability study indicated that reusing the spent catalysts led to a decrease from 69.0% to 65.0% and finally 61.0% on subsequent cycles indicating some loss of activity over time. Post reaction characterization of the spent catalyst indicated that the loss of activity was due to carbon deposition as evidenced by FTIR and EDX data. This research contributes to the body of knowledge addressing the issue of visible light driven photocatalysis for the amelioration of recalcitrant environmental pollutants such as POME.

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LIST OF SYMBOLS

c	characteristic constant of the adsorbate
λ	Wavelength (nm)
θ	Bragg's angle of incidence
β_{obs}	width at half maximum intensity
β_{inst}	standard instrumental line width
β_{d}	a true line width at half maximum intensity
α	photocatalyst coefficient
V_{m}	volume of gas adsorbed in correspondence to the monolayer coverage
t	adsorbed layer of thickness
r_{K}	Kelvin radius of the pore
r_{COD}	COD degradation rate in ppm min ⁻¹
P_{a}	ambient pressure
OH^{\bullet}	hydroxyl radical
$\text{O}_2^{\bullet-}$	superoxide radical
k_{Sch}	Scherrer constant which assumes the numerical value of 0.93
K_{A}	adsorption equilibrium constant
$h\nu$	Incident light
H^+	protons
h^+	Photo-generated holes
g	photocatalyst mass
E_{bg}	Band gap energy
$d_{\text{-spacings}}$	inter-planar distances
D	crystalline size (nm)
C_{Ao}	initial COD concentration in the sample in ppm

LIST OF ABBREVIATIONS

AC	auto-combustion
AOPs	advanced oxidation processes
AV	accelerating voltage
BET	Brunauer-Emmett-Teller
BJH	Barrett, Joyner and Halenda analysis
BOD	Biochemical Oxygen Demand
BTSE	biologically treated secondary effluent
CB	conduction band
COD	Chemical Oxygen Demand
CP	co-precipitation
CPO	Crude Palm Oil
CSPO	Certified Sustainable Palm Oil
CSTR	continuous stirred tank reactors
DH	Dollimore and Heal analysis
DOE	Malaysia Department of Environment
DRS	Diffuse Reflectance Spectroscopy
E Coli	Escherichia coli
EDX	Energy Dispersive X-Ray Microanalysis
EFB	empty fruit bunch
EGSB	expanded granular sludge bed
EU	European Union
GHG	greenhouse gas
HEGM	hexane extractable gravimetric method
HRT	Hydraulic Retention Time
ICDD	International Centre for Diffraction Data
IPA	isopropyl alcohol
LH	Langmuir-Hinshelwood
LUMO	lowest unoccupied molecular orbital
MABB	modified anaerobic baffled bioreactor
MOF	metal organic framework
MSPO	Malaysian Sustainable Palm Oil

NGO	Non-Governmental Organization
O&G	Oil and Grease
OFAT	One Factor at a Time
OLR	organic loading rate
OPKS	oil palm kernel shells
PAC	polyaluminum chloride
PL	Photoluminescence Spectroscopy
POME	Palm Oil Mill Effluent
PTFE	polytetrafluoroethylene
ROS	reactive oxygen species
RSPO	Roundtable of Sustainable Palm Oil
SCS	solution combustion synthesis
SEM	Scanning Electron Microscopy
SKM	Schuster-Kubleka-Munk Model
SS	Suspended Solids
SSR	solid state reaction
TPA	terephthalic acid
TSS	Total Suspended Solids
UAMAS	ultrasonic-assisted membrane anaerobic systems
UASB	up-flow anaerobic sludge blanket
UASFF	up-flow anaerobic sludge fixed film
US	microwaves, ultrasonic
UV	ultra violet
VB	valence band
VFAs	volatile fatty acids
WWF	World Wildlife Foundation

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