PHOTOCATALYTIC DEGRADATION OF PALM OIL MILL SECONDARY EFFLUENT (POMSE) USING ZINC OXIDE NANOPARTICLES

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This thesis dedicated to my beloved husband, father, mother, daughter, family and to whom that I love for their endless support.

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ABSTRACT

Palm oil mill secondary effluent (POMSE) properties still does not achieve the discharged requirement by the department of environment (DOE). Hence, it would be a solution to the POMSE treatment to recover and reuse the photocatalyst as to meet the development of greener and advanced technologies. However, the study on POMSE treatment using photocatalysis process in presence of ZnO-PEG nanoparticles and its physicochemical is still limited. Therefore, this study reports on the comparison of physicochemical properties between the ZnO nanoparticles in presence of the different type of capping agent Commercial ZnO, ZnO-PEG and ZnO-PVP, photocatalysis of POMSE by using ZnO nanoparticles and their potential of their reusability. The physicochemical of the ZnO nanoparticles had been analysed using XRD, FTIR and TEM where the results show that there are no impurities present in the samples and presenting the nature and chemical bonds for ZnO-PEG, besides having less agglomeration and smaller average in size (25-150 nm) compared to Commercial ZnO and ZnO-PVP. ZnO-PEG nanoparticles have a great potential in degradation of POMSE and this is supported with the results evaluated from four potential factors which are (A) different type of photocatalysts, (B) initial pH of the POMSE, (C) loading of ZnO-PEG and (D) concentration ratio of POMSE. It was found that all the four main factors were substantial, with contributions of (A) 73%, (B) 73 %, (C) 84% and (D) 84% respectively, to the POMSE degradation. Accordingly, the most favourable condition for the photocatalysis process of POMSE is under pH 6.5 in presence of ZnO-PEG with 0.5 g/L for the 25% of concentration ratio of POMSE dilution. The calcination methods portrayed the maximum degradation of POMSE colour after second use by 74% colour removal. Besides, the same molecular components and structures for XRD and FTIR were portrayed which indicates the reusability method is performed well.

ABSTRAK

Effluen kedua minyak kelapa sawit (POMSE) masih belum mencapai keperluan yang ditatpkan oleh jabatan alam sekitar (DOE). Penyelidikan ini akan menjadi penyelesaian kepada rawatan POMSE untuk memulihkan dan menggunakan semula fotopemangkin seiring dengan perkembangan teknologi hijau dan canggih. Walau bagaimanapun, kajian mengenai rawatan POMSE menggunakan proses fotopemangkin dengan kehadiran nanopartikel ZnO-PEG dan fizikokimianya masih terhad. Oleh itu, kajian ini melaporkan perbandingan fizikokimia antara tiga jenis nanopartikel ZnO; ZnO Komersial, ZnO-PEG dan ZnO-PVP, fotopemangkinan POMSE dengan menggunakan nanopartikel ZnO dan potensi penggunaan semula. Fizikokimia nanopartikel ZnO telah dianalisa menggunakan XRD, FTIR dan TEM di mana hasilnya menunjukkan bahawa tidak terdapat sebarang benda asing di dalam sampel dan mempamerkan ikatan semulajadi dan kimia untuk ZnO-PEG, selain mempunyai aglomerasi yang kurang dan purata saiz yang lebih kecil (25-150nm) berbanding dengan ZnO Komersial dan ZnO-PVP. ZnO-PEG nanopartikel mempunyai potensi yang besar dalam penguraian POMSE dan ini disokong oleh penilaian dari empat faktor yang berpotensi iaitu A) jenis fotopemangkin, (B) pH awal POMSE, (C) pemuatan ZnO-PEG dan (D) nisbah kepekatan POMSE telah dinilai. Hasil kajian mendapati bahawa semua empat faktor utama adalah signifikan dengan sumbangan (A) 73%, (B) 73%, (C) 84% dan (D) 84% terhadap penguraian POMSE. Oleh itu, keadaan terbaik bagi proses penguraian POMSE adalah di bawah pH 6.5 dengan kehadiran ZnO-PEG dan 0.5 g / L untuk 25% nisbah kepekatan POMSE. Kaedah kalsinasi menunjukkan degradasi maksimum warna POMSE selepas penggunaan kedua dengan penyingkiran berwarna sebanyak 74%. Selain itu, XRD dan FTIR menggambarkan komponen dan struktur molekul yang sama mebuktikan kaedah reusability dilaksanakan dengan baik.

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LIST OF SYMBOLS AND ABBREVIATION

Ag	-	Silver
AnPOME	-	Anaerobically treated palm oil mill effluent
Au	-	Gold
AuNPs	-	Gold nanoparticles
BOD	-	Biochemical oxygen demand
BOD_5	-	BOD value from the 5 days' test
C	-	Concentration of POMSE
C	-	Celsius
C_0	-	Concentration of POMSE at time zero
Ca	-	Calcium
$C_aH_bO_c$	-	Complex organic pollutant
CB	-	Conduction band
CdS	-	Cadmium sulphide
CH_4	-	Methane
CO_2	-	Carbon dioxide
COD	-	Chemical oxygen demand
CuO	-	Copper oxide
D_1	-	DO of the prepared samples immediately after preparation
D_2	-	DO of the prepared sample after incubation of 5 days
DAF	-	Dissolved air floatation
df	-	Dilution factor
DO	-	Dissolved oxygen
DOE	-	Department of Environmental

e^-	-	Negative electrons
EFB	-	Empty fruit bunch
EGSB	-	Expanded granular sludge bed
EQA	-	Environmental quality act
eV	-	Electro Voltage
Fe_2O_3	-	Iron (II) oxide
FFB	-	Fresh fruit bunch
FTIR	-	Fourier transform infrared spectroscopy
h+	-	Positive holes
H_2O	-	Water
H_2O_2	-	Hydrogen peroxide
HCl	-	Hydrochloric acid
HRT	-	Hydraulic retention time
IR	-	Infrared
K	-	Potassium
K	-	Kelvin
kV	-	Kilo Volt
meV	-	Mega-electron volt
Mg	-	Magnesium
MgO	-	Magnesium oxide
MLSS	-	Mixed liquor suspended solids
MPOB	-	Malaysian Palm Oil Board
MPR	-	Membrane photocatalytic reactor
MT	-	Membrane technology
M_w	-	Molecular weight
N	-	Nitrogen
$NaBH_4$	-	Sodium borohydride
NaOH	-	Sodium hydroxide
NHCOR	-	Amides groups
NR_2,	-	Groups
O_2	-	Oxygen
O_2^-	-	Super-oxide anions
OCOR	-	Esters groups

OH	-	Hydroxyl groups
$OH^{\bullet}$	-	Hydroxyl radical
OR	-	Alkyl groups
P	-	Phosphorus
P	-	Decimal volumetric fraction of sample used
PAA	-	Polyacrylic acid
PCA	-	Photocatalytic activity
PEG	-	Polyethylene glycol
PEO	-	Polyethylene oxide
POE	-	Polyoxyethylene
POME	-	Palm oil mill effluent
POMSE	-	Palm oil mill secondary effluent
ppb	-	Part per billion
PPME	-	Paper mill effluent
PVP	-	Polyvinylpyrrolidone
ROS	-	Reactive oxygen species
SEM	-	Scanning electron microscope
SI	-	Scum index
SVI	-	Sludge volume index
TEM	-	Transmission electron microscopy
TiO_2	-	Titanium oxide
UIRL	-	University Industries Research Laboratory
UTHM	-	Universiti Tun Hussein Onn Malaysia
UV	-	Ultraviolet
VB	-	Valence band
VOC	-	Volatile organic compound
WO3	-	Tungsten oxide
Wz	-	Wurtzite
XRD	-	X-ray diffraction
Zn^{2+}	-	Zinc cation
ZnO	-	Zinc oxide
ZnO-PEG	-	ZnO in the presence of PEG capping agent
ZnO-PVP	-	ZnO in the presence of PVP capping agent

ZnO-PVP-St	-	ZnO under stirring condition in the presence of PVP
ZnS	-	Zinc sulphide
ZPC	-	Zero-point charge

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CHAPTER 1

INTRODUCTION

1.1 Background

Malaysia is well acknowledged as the second major producer of palm oil after Indonesia. Despite this main industry has a big contribution to the economy, they had generated other major disposal problem in disposing of all the by-products and the palm oil mill effluent (POME). POME has been acknowledged as one of the major sources of water pollution in Malaysia due to the ensuing high biochemical oxygen demand (BOD) and chemical oxygen demand (COD) (Darajeh *et al.*, 2016). The treatment of POME in Malaysia generally will go through a few stages of treatment such as ponding system, open tank digester, and prolonged aeration system, or secure anaerobic digester and land application system (Wu *et al.*, 2010). The pre-treated POME or palm oil mill secondary effluent (POMSE) is the product after biological treatment of POME and is considered as a thick, brownish colour, bad odor, higher pH (7 to 9 pH) but has a lower BOD and COD compared to POME (Shahrifun *et al.*, 2015). Even though some approaches had been conducted to minimize the environmental threat, which is by using biological treatment, the brownish colour of the pre-treated POME still cannot be diminished.

Researchers had emanated that due to its high volume generated, POMSE is the hardest waste to handle all the waste produced (Madaki & Lau, 2013). The large quantity of POMSE in water stream might lead to oxygen depletion in water stream as POMSE comprises high amount of nitrogen (N), phosphorus (P), potassium (K), magnesium (Mg), and calcium (Ca) which will boost the growth of the plant in the water region (Embrandiri, Ibrahim, & Singh, 2013). Gobi and Vadivelu (2013) declared that the anaerobic and aerobic (open tank digestion and extended aeration) treatment systems are deliberated as obsolete and faulty as POMSE is not treated effectively. In addition, Ahmad *et al.,* (2003) initiated that the futile of the treatment is because of the sensitivity of the microorganism existing towards the temperature changes and pH fluctuation. Other than that, high organic content, silting, excessive sludge, need lots of labour, involving a massive space and land, producing methane as biogas, time-consuming hydraulic retention time (HRT) and also short-circuiting in aerobic and anaerobic treatment may be the encounters to reach the standard discharge limit (Abdullah & Sulaiman, 2013; Madaki & Lau, 2013). Yet, this type of treatment is still the highest selectable because it is cost-effective. Therefore, one of the promising approaches that can be implemented in order to degrade the colour of POMSE is by using photocatalytic degradation process (Alhaji *et al.,* 2016).

During photocatalytic degradation process, the hazardous organic chemical will be mineralized to become water, carbon dioxide and simple mineral acids (Akpan & Hameed, 2009). Previously, Mondal & Sharma (2016) claimed that photocatalytic degradation is one of the promising methods due to their capability for fully mineralizing the organic contaminants under mild conditions such as ambient temperature and pressure. Beydoun, Amal, Low, & McEvoy (1999) supported this notion by stating the advantage of the photocatalysts used in photocatalysis processes is they can be reused or recycled by their self-generated. In addition, among all the methods for photocatalytic degradation of dyes, ultraviolet (UV) irradiation had been acknowledged for its effectiveness in remove the pollutants and the environmental purification (Tripathy *et al.,* 2014).

In 2009, Yogamalar, Srinivasan, & Bose indicated that the addition of capping molecule could greatly influence the size confinement of ZnO nanostructures, which in turn affects the physical properties. A significant size variation of the particle is noted with the addition of capping molecules. The addition of capping molecules also confined the particle to a greater extent. Recently, Desa *et al.,* (2017) and Chiranont *et al.,* (2017) reported that ZnO-PVP and ZnO-PEG nanoparticles have a great potential as a photocatalyst for industrial dye wastewater treatment. This is due to the maximum degradation of dyes in a short period which is around two minutes. However, there is no physicochemical study of ZnO-PVP and ZnO-PEG nanoparticles and their reusability in photocatalytic degradation of dyes. It is almost impossible to reuse the photocatalyst again in the photocatalysis process by using the conventional method of wastewater treatment. This would be an advantage for the coupling method of photocatalysis process and membrane to separate and reutilize back the photocatalysts. Hence, it is very important for the industries as they can lower their chemical usage besides reducing their cost.

There were few types of research concerning the reusability of a photocatalyst in photocatalysis process. Most of them were succeeding to reutilize the photocatalyst back in the remediation of the wastewater with a slightly reduced performance after a few cycles of the consecutive run. For instance, Velmurugan *et al.*, (2011) had conducted three consecutive runs in order to study the photocatalyst reusability which is the ZnO nanocrystals. The photocatalyst did show a remarkable photostability degraded around 85.8% of dye after the third run. Other than that, Gomez *et al.*, (2015) had been reutilizing the photocatalyst by filtering, washed, dried in air oven under 70^oC. Despite that, the result did not portray a large difference in term of degradation percentage nor the mineralization for the 1^{st} and the 8^{th} cycles. Nevertheless, there is no previous study regarding the reusability potential of a photocatalyst for POMSE.

1.2 Problem Statement

Previously, (Hanis *et al.*, 2014) confirmed that ZnO under stirring condition in the presence of PVP (ZnO-PVP-St) via precipitation method has the great performance in membrane photocatalytic reactor (MPR). However, the effect of PEG as capping agent for producing ZnO-PEG-St had not been testified. There are very limited details studies involving the effect of capping agent in the synthesizing of the ZnO nanoparticles via precipitation method in term of their performance in degrading colour and their physicochemical properties. To acquire a better performance of photocatalyst, the ZnO nanoparticles must obtained smaller without any agglomeration by addition of capping agent during the synthetization. PEG could act a bridge-linking role due to its multiple coordinating sites and its linear structure. Hence it is effectively adsorbed on the ZnO nanoparticles' surfaces (Wang *et al.*, 2007). PEG steered a big role in congregating and altering the morphology of the ZnO nanoparticles due to its steric interference.

On top of that, there are some major limitation concerning the recovery of the photocatalyst in photocatalysis process. Recently, (Desa, 2017) and (Chiranont, 2017) reported that ZnO-PVP and ZnO-PEG nanoparticles have a great potential in photocatalytic degradation of industrial dye wastewater. However, the physicochemical study of ZnO-PVP and ZnO-PEG nanoparticles and their reusability in photocatalysis process had never been observed.

Palm oil mill secondary effluent (POMSE) has high colour intensity, dissolve oxygen (DO), turbidity, and organic load of BOD which still not achieved the discharged requirement by the department of environment and led to detrimental condition to the aquatic life (Wu *et al.*, 2010). Photocatalytic degradation process is one of the promising methods in wastewater treatment due to its advantages. However, the study on POMSE treatment using photocatalytic degradation process in presence of ZnO- PEG nanoparticles is still limited.

Therefore, this study aims to elucidate the photocatalytic activity and reusability of ZnO nanoparticles in treating POMSE as compared to commercial ZnO. This could be a preliminary and supported study for the next run in hybrid membrane photocatalysis reactor. In addition, the physicochemical properties of ZnO-PVP, ZnO-PEG, and commercial ZnO were explored in order to study the relationship of the photocatalysts morphology with their efficacy of reusable capability. This study would provide a solution to the industrial wastewater treatment to recover and reuse the photocatalyst as well as reduction of the chemical usage in order to meet the development of advanced and greener technologies. The regeneration of the photocatalyst is very crucial to reduce the cost and effectiveness of the photocatalytic process.

1.3 Research Objectives

The objectives of this research are:

(i) To identify the effects of different types of capping agents on the physicochemical properties of ZnO nanoparticles.

(ii) To evaluate the effects of operating parameters on the photocatalytic degradation efficiency of the synthesized photocatalyst on POMSE.

(iii) To determine the effects of different reusability methods on the physicochemical properties and performance of the photocatalyst.

1.4 Scope of Research

The scopes of this research are:

(i) Synthesis of ZnO nanoparticles via precipitation method by using different types of capping agents (PEG and PVP)

(ii) Characterization of the ZnO nanoparticles in term of their physicochemical properties using transmission electron microscopy (TEM), X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR).

(iii) Elucidation of the photocatalytic activity and reusability of the ZnO photocatalyst for POMSE:

   a. Photocatalytic activity of ZnO nanoparticles under various pH (4-10), different photocatalysts (ZnO-PVP, ZnO-PEG, and Commercial ZnO), various loading (0.08-0.9 g/L), and various concentration ratio of the POMSE (0-75% dilution).
   b. Reusability of ZnO photocatalysts by using a different method: (1) Oven drying method and (2) Calcination method (3) Characterization of reusable photocatalysts
   c. Analysis of the treated wastewater in terms of colour intensity, turbidity, dissolved oxygen (DO), biochemical oxygen demand (BOD) and chemical oxygen demand (COD).

1.5 Significance of Study

This research would be beneficial for the palm oil mill industry to improve the environmental performance by maximizing water quality via photocatalysis process. Therefore, it is parallel with the national agenda that emphasised on the preservation and conservation of the environment. This is a preliminary study before undergoing to the next stage which is membrane photocatalytic reactor as this coupling system would enable the photocatalyst to be collected back from the previous run. Besides, this study can compare the best method to recover and reuse the photocatalyst as well as reduction of the chemical usage to meet the development of advanced and gree technologies.

CHAPTER 2

LITERATURE REVIEW

2.1 Oil palm wastes

Oil palm is the essential product from Malaysia that aided to the transformation of the scenario of the agriculture and economy. It was shown by the dramatic growth of the oil palm planted in Malaysia from a mere 55 000 hectare in 1960 to 193 000 hectares in 1970 (Nambiappan *et al.*, 2018). The expansion was exceptional with the planted area achieving 1.02 million hectares in 1980 and successively reaching to 2.03 million hectares in 1990 and further to 5.74 million hectares in 2016 (MPOB, 2017). The statically value as depicted in Figure 2.1. The vast growth of the oil palm circa 1960s and 1970s resulting in the conversion of the land from rubber to oil palm due to the downtrend of the rubber price. Furthermore, the oil palm was proven more lucrative than rubber as well as having a low cost of transition and the similar infrastructure requirement with the rubber. Consequently, oil palm planted enormously in estates along with the launching of new land areas particularly under the government schemes (Arshad, 2007).

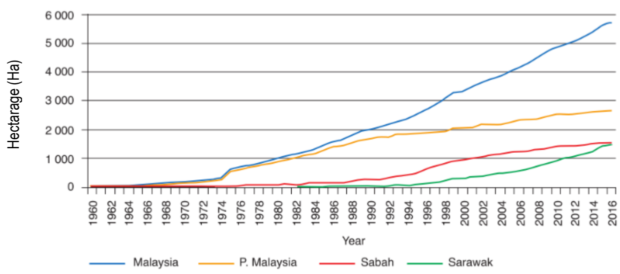

Figure 2.1: Statistical value for the oil palm planted area in Malaysia (MPOB, 2017)

The production of palm oil in Asia is conquered by Indonesia, Malaysia, and Thailand, together accounting for circa 95% of Asia's total palm oil production (Abu Bakar *et al.*, 2017). Regardless of being partly responsible to the agricultural and economic scenario, palm oil mill also significantly leads to environmental damaging and being an issue of major concern today (Abdullah & Sulaiman, 2013; Embrandiri *et al.*, 2013). There are about 430 palm oil mills in Malaysia that produce about 18.9 million tonnes of crude palm oil obtained from 92.9 million tonnes of fresh fruit bunches, with the assumption that the ratio of fresh fruit bunches processed to POME generated is 1:1.5 (Wahab *et al.*, 2013).

Oil palm waste is also produced from the refined fresh fruit bunch (FFB) which comprises of 3% decanter cake, 6% shell, 28.5 % empty fruit bunch (EFB) and 30% fiber (Sia, Tan, & Abdullah, 2017). The mills are most often situated in the estates and the prevailing practice is assembling the waste and discarding them in the most intuitive manner as excess nutrients may be detrimental to both the growing plants and the ecosystem overall. Even though some researchers had proposed alternative sustainable management, there is still much work to be done as the quantities being produced daily surpasses its use. Environmental- friendly approaches are obligatory right from the reaping to the process of the mills such as zero burning, waste minimization and reusing or recycling of the wastes (Embrandiri, Ibrahim, & Singh, 2016).

2.2 Palm oil mill effluent (POME)

Processing of palm oil fruit for extraction of oil can be achieved by a combination of different methods such as sterilization, stripping, digestion, clarification, and stripping. Most of these methods involve excessive use of water. Chang (2014) had claimed that 1.5 m^3 of water are needed to process one tonne of FFB of palm fruit and 50% of this volume ends up as palm mill oil effluent (POME) which consequently becomes a remarkable threat to water environmental system. Based on Figure 2.2, it has been proven POME contributed about 67% of the total wastes from the FFB processing. It is the major waste contributor in the palm oil mills. It is also acknowledged as one of the most polluting agro-industrial residues due to its high organic load. It holds a variety of suspended components including an assembly of minor organic and mineral

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