SYNTHESIS AND APPLICATION OF POLYACRYLAMIDE GRAFTED MAGNETIC CELLULOSE FLOCCULANT FOR WASTEWATER TREATMENT

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Dedicated specially to my parents, siblings and all of my friends.
Without whom none of my success would be possible

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ABSTRACT

A continuous presence of organic loads in palm oil mill wastewater treatment plants has lately risen as a major issue. This situation shows that coagulation-flocculation process should be overhauled in removing micro-size particles from palm oil mill effluent. Therefore, a direct flocculation process was proposed as pre-treatment to remove total suspended solid (TSS), turbidity, chemical oxygen demand (COD) and colour. In this study, a new polyacrylamide grafted onto magnetic cellulose extracted from oil palm empty fruit bunch (PAM-g-mcOPEFB) was successfully synthesised by using a microwave assisted synthesis method. The physical and chemical properties of PAM-g-mcOPEFB was characterized by using Fourier transform infrared spectroscopy, vibrating sample magnetometer, zeta potential and elemental analysis. A jar test method was employed to perform direct flocculation. The flocculation studies have been evaluated on anaerobically treated palm oil mill effluent suspension. Important parameters including flocculants dosage, pH of solution and settling time were varied to study the effects of these independent variables. For grafting synthesis study, the highest grafting percentage was achieved at 155.25%. It was observed that the best condition for the reduction of TSS, turbidity, COD, and colour were achieved at flocculants dosage of 1.5 g/L, pH of 8.0 and settling time of 30 minutes. Under this condition, the reduction of TSS, turbidity, COD, and colour was 82.97%, 88.62%, 53.23% and 91.76%, respectively. Prepared PAM-g-mcOPEFB also showed better performance when compared with cellulose, magnetic cellulose (mcOPEFB), polyacrylamide, and alum. In conclusion, direct flocculation by utilizing PAM-g-mcOPEFB can possibly be one of the best alternative flocculants to remove small particles of organic matters in palm oil mill treatment plants due to its simplicity and environmentally friendly method.

ABSTRAK

Kewujudan kandungan organik yang berterusan di loji rawatan air buangan kilang kelapa sawit telah muncul sebagai isu yang serius semenjak kebelakangan ini. Keadaan ini telah menunjukkan bahawa proses pengentalan-pemberbukuan perlu ditingkatkan bagi penyingkiran partikel bersaiz mikro dari efluen kilang kelapa sawit. Oleh itu, proses pemberbukuan secara langsung dicadangkan sebagai pra-rawatan untuk menyingkirkan total pepejal terampai (TSS), kekeruhan, permintaan oksigen kimia (COD) dan warna. Dalam kajian ini, poliakrilamida tercantum selulosa magnet yang diekstrak daripada tandan kelapa sawit kosong (PAM-g-mcOPEFB) yang baharu telah berjaya disintesis dengan menggunakan kaedah sintesis berbantukan gelombang mikro. Sifat fizikal dan kimia PAM-g-mcOPEFB telah dicirikan dengan menggunakan spektroskopi inframerah jelmaan Fourier, magnetometer sampel bergetar, potensi zeta dan analisis elemen. Pemberbukuan secara langsung dilakukan dengan menggunakan kaedah ujian balang. Kajian pemberbukuan dijalankan kepada efluen kilang kelapa sawit dirawat secara anaerobik. Parameter penting termasuk dos bahan berbuku, pH larutan dan masa penetapan telah diubah untuk mengkaji kesan parameter-parameter tersebut. Di dalam kajian mengenai sintesis pencatuman, peratusan pencantuman tertinggi dicapai sebanyak 155.25%. Keadaan terbaik bagi pengurangan TSS, kekeruhan, COD dan warna dicapai pada dos bahan berbuku 1.5 g/L, pH 8.0 dan masa penetapan 30 minit. Dalam keadaan ini, pengurangan TSS, kekeruhan, COD dan warna masing-masing adalah 82.97%, 88.62%, 53.23% dan 91.76%. PAM-g-mcOPEFB yang dihasilkan juga menunjukkan prestasi yang lebih baik jika dibandingkan dengan selulosa, selulosa magnet (mcOPEFB), poliakrilamida dan alum. Kesimpulannya, pemberbukuan secara langsung yang menggunakan PAM-g-mcOPEFB berpotensi menjadi salah satu alternatif bahan berbuku terbaik untuk menyingkirkan partikel kecil di dalam bahan organik di loji rawatan kilang kelapa sawit dengan cara yang mudah dan mesra alam.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AGU	–	Anhydroglucose unit
AnPOME	–	Anaerobically treated Palm Oil Mill Effluent
AN	–	Anaerobic pond
AR	–	Aerobic pond
ASTM	–	American Society for Testing and Materials
BOD	–	Biochemical oxygen demand
CAN	–	Ceric ammonium nitrate
CAS	–	Ceric ammonium sulphate
COD	–	Chemical oxygen demand
CPO	–	Crude palm oil
DOE	–	Department of Environment
EA	–	Elemental analysis
EDX	-	Energy-dispersive X-ray
EFB	–	empty fruit bunch
FAS	–	Ferrous ammonium sulphate
FRIM	–	Forest Research Institute Malaysia
FTIR	–	Fourier Transform Infrared Spectroscopy
GA	–	glutaraldehyde
GNI	–	gross national income
HRT	–	hydraulic retention time
MPOB	–	Malaysian Palm Oil Board
MPOC	–	Malaysian Palm Oil Council
MION	–	Magnetic iron oxide nanoparticles
MNPs	–	Magnetic nanoparticles
NA	–	not available

PAA	–	Polyacrylic acid
PAC	–	Polyaluminium chloride
PAM	–	Polyacrylamide
POME	–	palm oil mill effluent
PORIM	–	Palm Oil Research Institute of Malaysia
SBR	–	Sequencing batch reactor
SVI	–	sludge volume index
TN	–	Total nitrogen
TOC	–	Total organic carbon
TSS	–	Total suspended solid
VSM	–	Vibrating sample magnetometer

LIST OF SYMBOLS

°C	–	degree celcius
%G	–	grafting percentage
X_i	–	initial reading of parameters
X_f	–	final reading of parameters
emu/g	–	electromagnetic unit per gram
g/L	–	gram per Liter
mg/L	–	milligram per Liter
kg	–	kilogram
km	–	kilometer
m	–	meter
m^3	–	cubic meter
mA	–	miliAmpere
mL	–	milliliter
mV	–	millivolts
ppm	–	parts per million
PtCo	–	platinum cobalts
R	–	squareness
rpm	–	rotation per minute

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter will briefly present the introduction on wastewater from palm oil industry that contains various types of organics and inorganics. The research on new development of treatments that might help treat the environment by the effluents that produce negative impacts is carried out. The problem statement, objectives and scope of research are also highlighted.

1.2 Research Background

The oil palm is very familiar industry in Malaysia as oil palm plantations can be found throughout the country. Some of the responsible agencies for the success of this industry include Malaysian Palm Oil Board (MPOB), Malaysian Palm Oil Council (MPOC), Palm Oil Research Institute of Malaysia (PORIM), Forest Research Institute Malaysia (FRIM), and some local universities (Bello and Abdul Raman, 2017). The

oil palm is the main produce for economic growth in Malaysia, making the country a role model in this industry for its nearby countries such as Indonesia, the Philippines and Thailand. Malaysia is undeniably a central source of this product. The oil palm industry contributed over $22.31billion USD to the Malaysia's gross national income (GNI) in 2014, making it as the fourth largest source of national earnings. In addition, over the past 30-40 years, the rapid increment of oil palm in Malaysia has changed the rural landscape in complex and highly contentious ways, generating a lot of economic, social and environmental issues as discussed in the literature.

While the supply of the palm oil has been expanded by the upgrades that have been concentrated, the results will likely be expanded. In palm oil industries, water pollution would be the considerable impact which should be dealt with so that the contamination could be diminished and controlled. Liquid waste or generally known as palm oil mill effluent (POME) contributes to a total of 58.4% of waste generated per ton of fresh fruit bunch processed in the industry (Steyer *et al.*, 2006). POME creates negative environmental impacts as it is 100 times more polluting than domestic waste, highly acidic and does not meet regulatory standards for direct discharge (Januri *et al.*, 2014). Compared to the receiving water body, POME consists of relatively high chemical oxygen demand (COD) and suspended solid content. In some reports, the approximate value of COD and suspended solid of biologically treated POME finally discharged are 520 and 217 mg/L at pH 8.5 (Othman *et al.*, 2014) which still do not meet the regulatory standard discharge limit of POME made by the Department of Environment (DOE). Thus, before discharged to the environment, it is better to reduce the contaminants of the biologically treated POME final effluent.

From the earlier stage of palm oil mill industry, ponding system is being used as the conventional method to treat POME for its low costs of construction, maintenance, and operation (Banat *et al.*, 1990), and can handle fluctuating organic hydraulic loads efficiently (Oswald, 1973; Oswald *et al.*, 1978). Apart from that, physical-chemical treatment (e.g. activated carbon, adsorption, coagulation/flocculation, membrane technology etc.) is also an alternative to overcome conventional POME treatment's drawbacks (Ahmad *et al.*, 2006; Ali Amat

et al., 2015). However, due to immense capital investment and insignificant income generation from the operation, and expensive maintenance, not many of the techniques have been employed at industrial or pilot scale (Alhaji *et al.*, 2016). In order to get rid of suspended and dissolved solids, colloids, and organic matter present in industrial wastewater, coagulation and flocculation is the most widely used solid-liquid separation process (Renault *et al.*, 2009).

Magnetic nanoparticles are the focus in water treatment field with the rapid evolvement of nanotechnology knowledge as magnetic nanoparticles (Fe_3O_4) are believed to show the finite-size impact and high proportion of surface-to-volume, achieving higher adsorption capacity (Mohammed *et al.*, 2017). Moreover, utilizing an external magnetic field can refine simple division of stacked magnetic nanoparticles from solution and enhance settling speed. Magnetic particles might adsorb contaminations from watery or gaseous effluents and this can be isolated through the simple magnetic process. There have been many reports on magnetic Fe_3O_4 particles coated with organic materials as adsorbent for metal removal (Bée *et al.*, 2017). In addition, the property of magnetic nanoparticles as coagulant has been reviewed to improve coagulation by the adsorption and magnetic effect of Fe_3O_4 nanoparticles.

1.3 Problem Statement

The primary environmental issues of palm oil industry are related to the water contamination resulting from indiscriminate discharge of non-treated POME into public watercourse. POME contains about 4-5% of total solid, high COD and BOD levels which contribute to high Sludge Volume Index (sludge produce per volume of wastewater) (SVI). Coagulation-flocculation is the most common physico-chemical treatment in solid-liquid separation process due to its simplicity and efficiency, and has been extensively used to treat various types of wastewater including POME.

Although ponding system has been applied to treat the POME through a series of anaerobic or aerobic pond, however, these wastes still do not meet the demand proposed by the DOE therefore placing the traditional method of effluent treatment as an inefficient method (Iskandar *et al.*, 2018). These ponds acquire an extensive open area subsequently producing a foul stench and predicament in maintaining the liquor dissemination and biogas collection which results in harmful effect to the environment (Chin *et al.*, 1996; Ng *et al.*, 1987; Onyla *et al.*, 2001). Despite the disadvantages of the pond system, a series of shallow ponds have also been practiced to minimize the effect of POME to the environment. Nevertheless, this method also requires larger space and has longer hydraulic retention time (Chan, 1983).

Bio-flocculants have emerged to be a guaranteed alternative since they are abundantly available and biodegradable polymers. Despite the fact that bio-flocculants display promising flocculating efficiency, its future development is restricted by a few disadvantages. Natural polymers have short life span because its active components will biodegrade with time. Moreover, they are moderately effective and their application is restricted as a coagulant aid.

To overcome the drawbacks and in conjunction with development of nanoparticles, magnetic cellulose is proposed due to its large surface–to-volume ratio and easy for separation. However, flocculation takes longer time, and the flocs tend to float, thus leading to low effectiveness when nanoparticles are used solely (Saifuddin and Dinara, 2011). Approaches to graft between natural and synthesis polymers have also been accomplished by many researchers because of their benefits in treating wastewater. However, in conventional method of grafting, the percentages of monomer attached to the backbone of natural polymer are much lower and the process needs to be carried out under an inert atmosphere resulting in low performance while flocculation process is being carried out.

Thus, to fulfil the gap in the mentioned drawbacks, this study intends to synthesize magnetic cellulose extracted from oil palm empty fruit bunch fibre grafted with polyacrylamide (PAM-g-mcOPEFB) via microwave-assisted method. To study the flocculation performance, PAM-g-mcOPEFB is used to treat anaerobically treated palm oil mill effluent (AnPOME) and the efficiency is defined through percentage reduction of turbidity, TSS, colour, and COD. Instead of POME, AnPOME was used in this research because to allow anaerobic digestion to occur and the aim in this research is to reduce number of aerobic ponding system.

1.4 Research Objectives and Scopes

Based on the identified problem statements, this research is carried out based on the objectives and scopes stated below:

1. To characterize and synthesize magnetic cellulose extracted from oil palm empty fruit bunch fibre (mcOPEFB).

The synthesis of mcOPEFB is carried out via crosslinking method. Ratios of magnet to cellulose dosage was varied from 1:2 to 2:1 and glutaraldehyde (as crosslinker) volume are varied from 0 to 2 mL. The best ratio and glutaraldehyde volumes are selected based on the highest removal of turbidity, TSS, colour, and COD, after jar test is performed. The best synthesized mcOPEFB is characterized with FTIR, VSM, zeta potential, and elemental analysis.

2. To synthesize and characterize polyacrylamide grafted onto magnetic cellulose (PAM-g-mcOPEFB)

PAM is grafted onto magnetic cellulose with the aid of microwave-assisted method. The mass ratio between PAM to mcOPEFB was varied from 4:1 to 1:4. Meanwhile, mass of ceric ammonium nitrate (CAN) is studied from 0 to 1.5 g. The best percentage grafting (%G) is selected for FTIR, VSM, elemental analysis, and zeta potential.

3. To evaluate and study PAM-g-mcOPEFB efficiency as flocculants for AnPOME treatment.

The performance of synthesized flocculants is tested using jar test experiment where a few variables i.e. flocculants dosage (0.5 – 2.5 g/L), initial pH of AnPOME (5 – 9), and settling time (15 – 75 minutes). For jar test conditions, the temperature is set at room temperature, the mixing rate is fixed at 200 rpm for rapid mixing (3 minutes) and 30 rpm for slow mixing (15 minutes). The removals of turbidity, colour, TSS, and COD from AnPOME are measured to indicate the effectiveness of flocculants. To distinguish the superiority of graft copolymer, tests are also performed solely on PAM, cellulose, mcOPEFB and alum (conventional coagulant).

1.5 Significance of the Study

After the DOE implements the requirement for the discharge of effluent from the crude palm oil industry under the Environmental Quality (Prescribed Premises) (Crude Palm Oil) Regulations 1977, the typical problems faced by the oil palm mills in Malaysia are balancing environmental protection, the economic practicality, and

sustainable development. Many mills' discharges do not follow the standards specified by the DOE Malaysia (Iskandar *et al.*, 2018). This shows that improving the existing wastewater treatment plants has to be done to adhere to the effluent standards established by the authorities. Immediate implication of this research is readily observable. The treatment system can be significantly improved by applying chemical pre-treatment in the POME treatment system. In addition, objectives to reduce number of ponds together and retention time are also been focussed in this research. The current findings will evaluate the effectiveness of grafted flocculant in treating AnPOME.

1.6 Organization of Thesis

Prior to this section, a brief introduction to the current scenario of palm oil industry and its negative effect on the environment has been discussed. The research background also states the conventional inefficient POME treatment which leads to the current study on the new development of magnetic flocculants.

Chapter 2 consists of the review of previous studies in recent years including conventional POME treatment, coagulation-flocculation, cellulose, magnetic nanoparticles, and its application in separating organic compound. The last part of this chapter reviews the use of various coagulants or flocculants in AnPOME and POME treatment.

Chapter 3 defines the research experimental work consisting of a list of materials and experimental methods utilised in this study. It includes the methods of flocculant synthesis and methods to characterize the prepared flocculants in addition to the experimental procedures in flocculation process in series mode for each reaction parameter.

Chapter 4 focuses on the properties, and discusses effects of prepared flocculants towards removal of turbidity, colour, and total suspended solid, as well as chemical oxygen demand in AnPOME. The comparison between synthesized flocculants and conventional coagulant are also presented.

Lastly, Chapter 5 structures the final conclusions on the findings of the entire research and directions for future works are suggested for improvement.

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