SYNTHESIS AND CHARACTERIZATION OF OIL PALM TRUNK HEMICELLULOSE DERIVATIVES FOR COAGULATION/FLOCCULATION REMOVAL OF CATIONIC DYES

NORSALLIANA BINTI SHAARI

UNIVERSITI TEKNOLOGI MALAYSIA

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NORSALLIANA BINTI SHAARI

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This thesis I dedicated to my beloved husband, father, mother, siblings, lecturers and fellow friends. Thanks for your support and prayers. Without all of you I will never be able to finish this project.

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ABSTRACT

Oil palm biomass such oil palm empty fruit bunch, oil palm frond and oil palm trunk (OPT) are considered as the most abundant agrowastes that have the potential to be utilized as raw materials for production of value-added products. Thus, the objective of this study is to synthesize a new novel flocculant based on hemicellulose derived from OPT for cationic dye removal application. Two different flocculants were synthesized in this study: (a) hemicellulose etherified with chloroacetic acid (HcECA) and (b) hemicellulose etherified with acrylamide (HcEAM). Both hemicellulose derivatives were prepared with various etherification mole ratios. The native hemicellulose and its derivatives were characterized using gel permeation chromatography, Fourier transform infrared and proton nuclear magnetic resonance. The coagulation/flocculation performance of the hemicellulose derivatives were evaluated with cationic dye (i.e. methylene blue, MB) at different initial pH values, hemicellulose derivative dosages and initial dye concentrations. The characterization results confirmed a successful synthesis of the respective hemicellulose derivatives with the improvement on the solubility of both derivatives. The coagulation/flocculation results indicated that HcECA15 has better ability to remove the cationic dyes compared to HcECA10 and HcECA5 whereas, HcEAM1 was better than HcEAM0.1, HcEAM0.5, HcEAM10 and HcEAM15. Changing the initial pH of the dye solution from acidic to alkaline resulted in the increase of dye removal percentage. In addition, higher dosages of hemicellulose derivatives also increased dye removal. On the contrary, high initial dye concentrations reduced the percentage of dye removal. HcEAM showed better efficiency on the cationic dye removal than HcECA due to its ability to produce denser flocs which are usually helpful during sedimentation. As a conclusion, both hemicellulose derivatives have their own novelty to be new flocculants for successful application in cationic dye removal processes. Yet, both flocculants had not fully decolourized the MB and still not highly likely to be applied in real industrial applications for textile wastewater treatment. Thus, the process, ratio of the etherification and mechanism of MB removal should be investigated further to enhance the dye removal performance.

ABSTRAK

Biojisim kelapa sawit seperti tandan buah kosong kelapa sawit, pelepah kelapa sawit dan batang kelapa sawit (OPT) dianggap sisa pertanian yang sangat berpotensi untuk memberikan nilai tambah kepada hasil pengeluaran. Maka, objektif kajian ini adalah untuk menghasilkan bahan pengelompokan yang baru iaitu hemiselulosa terbitan berasaskan OPT untuk aplikasi penyingkiran pewarna kationik. Dua jenis bahan pengelompok telah disintesis dalam kajian ini: (a) hemiselulosa di ester dengan asid kloroasetik (HcECA) dan (b) hemiselulosa diester dengan akrilamida (HcEAM). Kedua-dua hemiselulosa terbitan disediakan menurut nisbah mol pengesteran yang berlainan. Sifat hemiselulosa dan terbitannya telah dibuktikan menerusi kromatografi gel penelapan, inframerah transformasi Fourier dan resonans magnetik nuklear proton. Prestasi bahan pengelompok hemiselulosa terbitan dinilai melalui penyingkiran pewarna kationik (iaitu metilena biru, MB). Beberapa parameter telah ditetapkan semasa proses penyingkiran antaranya, nilai pH awal yang berbeza, dos terbitan hemiselulosa yang digunakan, serta kepekatan awal bahan pewarna. Keputusan ujian pencirian berjaya mengesahkan sintesis kedua-dua jenis hemiselulosa terbitan malah menunjukkan peningkatan dalam kebolehlarutan pada kedua-duanya. Keputusan pengentalan/pengelompokan menunjukkan, HcECA15 mempunyai keupayaan lebih baik untuk menyingkirkan pewarna kationik berbanding HcECA10 dan HcECA5. Manakala, HcEAM1 adalah lebih baik daripada HcEAM0.1, HcEAM0.5, HcEAM10, and HcEAM15. Selain itu, perubahan pH awal larutan pewarna daripada bersifat asid kepada alkali juga menyebabkan peningkatkan peratusan penyingkiran pewarna. Di samping itu, peratusan penyingkiran pewarna meningkat dengan pertambahan dos terbitan hemiselulosa. Walau bagaimanapun, peningkatan kepekatan awal pewarna menyebabkan penurunan peratusan penyingkiran pewarna. HcEAM menunjukkan kecekapan yang lebih baik ke atas penyingkiran pewarna kationik daripada HcECA kerana kemampuannya untuk menghasilkan kelompok padat yang membantu memudahkan proses pemendapan. Kesimpulannya, kedua-dua hemiselulosa terbitan mempunyai keaslian sebagai bahan pengelompok baru yang mampu memberi manfaat dalam proses penyingkiran pewarna kationik. Namun, kedua-dua bahan pengelompok masih belum dapat menyingkirkan keseluruhan MB dan masih tidak cukup kuat untuk diaplikasikan ke aplikasi industri sebenar untuk rawatan air sisa tekstil. Maka, proses, nisbah pengesteran dan mekanisma penyingkiran MB patut dikaji lagi bagi meningkatkan pencapaian penyingkiran pewarna.

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LIST OF ABBREVIATIONS

[AMIM][Cl]	-	1-ally-3-methylimidazolium chloride
[BMIM][Cl]	-	1-butyl-3-methyl-imidazolium chloride
^{1}HNMR	-	Hydrogen nuclear Magnetic Resonance
^{13}C NMR	-	Carbon Nuclear Magnetic Resonance
AB 1	-	Acid Black 1
Alum	-	Aluminium sulphate
AOX	-	Adsorbable Organic Halogens
AV 5	-	Acid Violet 5
BOD	-	Biological Oxygen Demand
CMC	-	Carboxyalkyl chitosan
CMS	-	Carbon molecular sieve
COD	-	Chemical Oxygen Demand
CR	-	Congo Red
CTS	-	Chitosan
D_2O	-	Dehydrated water
DADMAC	-	Diallydiamethyl ammonium chloride
DFS	-	Direct Fast Scarlet
DFY	-	Direct Fast Yellow
DO	-	Dissolved oxygen
DS	-	Dissolved solids
EFB	-	Empty fruit bunches
FFB	-	Fresh fruit bunches
FTIR	-	Fourier Transform Infrared Spectroscopy
GPC	-	Gel Permeation Chromatography
GY	-	Golden Yellow

HcECA	-	Hemicelluloses etherified with chloroacetic acid
HcEAM	-	Hemicelluloses etherified with acrylamide
HCl	-	Hydrochloric acid
H_2O	-	Water
H_2SO_4	-	Sulphuric acid
HPLC	-	High-performance liquid chromatography
ILs	-	ionic liquid
Ku-g-PAM	-	Ku (mucilage) grafting polyacrylamide
MB	-	Methylene Blue
MDF	-	Medium-density fire boards
MPOB	-	Malaysian Palm Oil Board
MF	-	Mesocarp fiber
NaOH	-	Sodium hydroxide
NB	-	Naphtalene Blue
NY	-	Nyanthrene Yellow
OPA	-	Oil palm ash
OPB	-	Oil palm biomass
OPF	-	Oil palm frond
OPKS	-	Oil palm kernel shell
OPT	-	Oil palm trunk
PACl	-	Polyaluminium chloride
PAFCl	-	Polyaluminium ferric chloride
PAM	-	Polyacrylamide
PAS	-	Polyaluminium sulphate
PDADMAC	-	Polydiallydimetyl ammonium chloride
PFCl	-	Polyferric chloride
PFF	-	Presses fruit fibre
PFS	-	Polyferrous sulphate
PKS	-	Palm kernel shell
POME	-	Palm oil mill effluent
PVA	-	polyvinyl alcohol
RB	-	Reactive Black
RB19	-	C.I Reactive Blue 19
RO5	-	C.I. Reactive Orange 5

RV	-	Reactive Violet
SACG	-	Self-adhesive carbon grains
SS	-	Suspended Solid
Tam-g-PAM	-	Tamar Indus Indica grafting polyacylamide
TDS	-	Total Dissolved Solid
UV	-	Ultraviolet

LIST OF SYMBOLS

C_e	-	Equilibrium concentration
C_o	-	Initial concentration
M_W	-	Weight-average molecular weight
M_N	-	Number-average

CHAPTER 1

INTRODUCTION

1.1 Research Background

Oil palm tree (*Elaeis guineensis*) is one of the most valuable plants in Malaysia, Indonesia and Thailand. One oil palm tree produce an average of 231.5 kg dry weight of biomass annually including the oil and lignocellulosic materials (Abdul Khalil *et al*., 2010c). In addition, oil palm industries contribute a few million tons of biomass per year. Solving the disposal problem is associated with good handling that involved creating value-added products from this biomass (Rozman *et al*., 2005).

Oil palm biomass (OPB) is an agricultural by-product, that was left in the field during the replanting, pruning and milling process of oil palm. OPB includes oil palm trunk (OPT), oil palm frond (OPF), oil palm kernel shell (OPKS), empty fruit bunch (EFB), pressed fruit fibre (PFF), and palm oil mill effluent (POME). Oil comprises only a small fraction of the total biomass produced by the plantation, while the remaining biomass is an abundant amount of lignocellulosic materials in the form of fronds, trunks and empty fruit bunch.

Therefore, oil palm industry must be prepared to take advantage of the situation and utilize the available biomass in the best possible manner (Basiron, 2007). The OPB is classified as a lignocellulosic residue which typically consists of 50% cellulose, 25% of hemicellulose and 25% of lignin in their cell wall (Alam *et al.*, 2009). Hemicellulose is a non-cellulosic heteropolysaccharides containing various units of sugar, arranged in different proportions and with different substituent (Glasser *et al*., 2000). Hemicellulose is a polymer with low molecular weight and branched with 80-200 degree of polymerization. The general formula is ($C_5H_8O_4$) also known as pentosan, and $(C_6H_{10}O_5)_n$ that is hexosan (Cai and Paszner, 1988).

Nowadays, there are a great deal of biological products being proposed and studied to develop effective coagulants and flocculants that would replace the conventional materials (Maximova and Dahl, 2006; Deng *et al.*, 2005; Chen and Lian, 2004; Jiang, 2001; Salehizadeh and Shojaosadati, 2001). Some of the reported named "bioflocculant" include biopolymers which are starch, chitosan, alginates and others (Wang *et al.*, 2007). In this study, hemicellulose is the biopolymer that was expected to make a novel flocculant or hemicellulose derivative that will be applied in the textile wastewater treatment. A hemicellulose derivative is a safe and biodegradable polymer that does not produce secondary pollution (Bratby, 2007; Sharma *et al.*, 2006; Crini, 2005; Arevalo, 2002; Hirohara *et al*., 1999).

Textile industry produces basic needs for people which are clothes (Módenes *et al*., 2012). Clothes often vary in pattern, style, colour and a few more characteristics. Therefore, textile industry involved multiple processes and consumed large amounts of water in the manufacturing process (Verma *et al*., 2012, Mishra *et al*., 2006; Anjaneyulu *et al*., 2005). Water is used mostly in the dyeing and finishing operations in which the fabric were given colour and processed into a commercialized product. As a result, a large volume of coloured effluents are highly generated from these activities. This polluted water should be treated before discharge to avoid negative environmental impacts (Jiang *et al*., 2011; Mishra *et al*., 2006).

The most important indicator of polluted water is the colour. The damaging effect is not only to the visual nature of the receiving streams, but the coloured contaminants are also toxic to aquatic life, carcinogenic, and mutagenic to humans (Jiang *et al*., 2011; Lucas and Peres, 2006; Hao *et al*., 2000; Liu and Qu, 2000). Currently, there are more than 100,000 dyes available in the textile industries around the world (Liu and Qu, 2000). Dye compounds are very stable and difficult to decolourize due to the different complex structures of dyes (Lucas and Peres, 2006; Liu and Qu, 2000).

The toxic effect and potential carcinogenic nature of dyes have increased the demand for a specific and cost-effective wastewater treatment unit that is more ecologically-friendly (Mishra *et al*., 2006). A former study by Anjeneyulu *et al.* (2005) listed a number of available methods for decolourization of dye such as adsorption, irradiation, ion exchange, ozonation, coagulation and precipitation, aerobic process, anaerobic process, single cell (fungal, alga, bacteria), advanced oxidation process, membrane filtration, photocatalysis, sonication, enzymatic treatment, redox mediators and engineered wetland system.

For many years, the main treatment or pre-treatment of dye-containing wastewater has been the coagulation process, due to the low capital cost (Anjaneyulu *et al*., 2005; Golob *et al*., 2005; Choi *et al*., 2001; Chu, 2001). However, there are some limitations in applying this method which related to the generation of sludge and ineffective decolourization of some soluble dyes (Anjaneyulu *et al*., 2005; Hai *et al*., 2007). Therefore, the effectiveness of the coagulation and flocculation in dye removal processes can be improved by proper selection of the coagulant/flocculant, and optimization of the process parameters including pH, initial concentration of dye or textile, dosage of coagulant/flocculants and others (Verma *et al*., 2012).

1.2 Problem Statement

Oil palm trees generate a large amount of biomass. In Malaysia, one hectare plantation of oil palm tree produces about 55 000 kgs of waste annually and 5 500 kgs of oil have been reported (Hasamudin and Soom, 2002). It is also reported that in 6 million hectares of plantation, about 11.9 million tons of oil and 100 million tons of biomass have been produced (Abdul Khalil *et al*., 2010b). As per mentioned in the research background, the average amount of biomass from an oil palm tree produced 231.5 kg dry weight per year (Abdul Khalil *et al*., 2010c). An estimation made by Malaysian palm oil industry stated that in 4.69 million hectare of plantation in which the production rate of dry oil palm biomass was 20.34 tons per hectare per year, 95.3 million tons lignocellulosic biomass were produced in 2009 (Lim, 1998).

Furthermore in late 2012, 80% of the world's total oil palm plantation area was monopolized by Malaysia and Indonesia, with exceeding 14 million hectares compared to other countries. The duration between growing, removing and replanting oil palm trees is 25 to 30 years. About 74 000 kg of dry waste palm wood per hectares are generated during replantation (Chin *et al*., 2012).

In order to implement the Zero Burning Policy on waste trunks in Malaysia's plantation, efforts were taken to utilize the biomass as an alternative to burning (Szymona *et al*., 2014). Oil palm trunks are commonly being left at the plantation to decompose on its own. However, oil palm trunks will eventually become a valuable part of the tree by turning the trunk wastes into a potential biomass.

Previous researchers managed to come out with the idea to exploit the trunks with some limitation due to its morphology and low mechanical properties (Tay *et al*., 2013; Prawitwong *et al*., 2012; Chin *et al*., 2010; Amouzgar *et al*., 2010). Therefore, a thorough investigation on biomass modifications should be done to effectively discard the massive amount of trunk wastes.

Oil palm trunk fibres are mainly composed of cellulose (41-42%), hemicelluloses (30-3l%), and lignin (14-16%). The trunks could potentially represent a very abundant, inexpensive, and renewable organic material. Economically viable products derived from the palm fibres are being eagerly sought and reconstituted into a number of technically feasible products (Gallacher *et al.*, 1994; Hassan *et al*., 1990).

Since hemicellulose can be found in natural low-cost sources like the oil palm trunk, the compound is expected to be one of the most promising coagulation/flocculation materials. Hemicellulose is potentially very useful (Lindblad *et al*., 2001). More often, the reported industrial applications for plant's hemicellulose include their use as viscosity modifiers, gelling agents, tablet binder or wet strength additives (Watson, 1959).

The use of hemicellulose derivatives as flocculants in dye removal processes requires modification or derivation. The modification or derivation of these polymers created novel opportunities to maximally exploit the various valuable properties of hemicellulose for previously unperceived applications (Ebringerová and Heinze, 2000). Modification methods towards enhancing a hemicellulose's solubility were obtained from a former study based on starch, cellulose and chitosan (Haack *et al*., 2002; Zhang, 2001; Thanou *et al*., 2000).

The preparation and properties of new polymers from hemicellulose are the valuable information for any research program aimed at utilizing annually renewable and agriculture-derived polymers. This present research has focused on synthesizing novel hemicellulose derivatives as flocculants for dye removal application to the textile wastewater treatment.

Batik industry is the biggest cottage textile industry in Indonesia and Malaysia especially in Kelantan and Terengganu. This homemade textile industry has making a big contribution to the economic growth due to high demands locally and from abroad.

However, previous studies by Ahmad *et al.* (2002) show that the wastewater from batik industry which containing grease, wax, heavy metal, surfactant, suspended solid, and dyes (organic and inorganic) caused a serious problem to the environment due to the manufacturer commonly discharge their effluents into environment without appropriate treatment.

Without proper treatments of the wastewater contained dye, the effluent became an ecological concern as the pollutants would bring acute and chronic effects to people, plants, and animals. Therefore, a safe and applicable solution is required to recover this problem. The flocculants in the coagulation/flocculation process may overcome the colour removal problem faced by the textile industry (Sharma *et al*., 2006).

Consequently, treatments on batik effluent pollution to the environment are very critical and get much attention from the researchers. Thus, this present research which focusing in synthesizing of novel hemicellulose derivatives as flocculants for dye removal application will be beneficial to the textile wastewater treatment especially for batik industry in Malaysia.

1.3 Objectives

1. To synthesis and characterize hemicelluloses derivatives
2. To study the dye flocculation performance of hemicelluloses derivatives
3. To compare both types of hemicelluloses derivatives

1.4 Scopes

1. Hemicelluloses derivatives were prepared using isolated hemicelluloses from oil palm trunk (OPT) to produce new novel flocculants for cationic dye (methylene blue) removal application. Two different novel flocculants were synthesized in this study: (a) hemicelluloses etherified with chloroacetic acid (HcECA) and (b) hemicelluloses etherified with acrylamide (HcEAM). Both hemicellulose derivatives were prepared with varies etherification mol ratios (hemicellulose: reagent). The native hemicellulose and its derivatives were characterized using gel permeation chromatography (GPC), Fourier transform infrared (FTIR) and hydrogen nuclear magnetic resonance (^{1}HNMR).

2. The performance of the flocculants were determined using Jar test method. The effects of etherification ratio of each hemicellulose derivatives, initial dye concentration, hemicellulose derivative dosages, and initial pH of the dye on dye coagulation/flocculation process were investigated using synthetic dye (methylene blue) solution and batik's dye wastewater. The dye concentration was measured using UV-Vis spectrophotometer. The percentage of dye removal indicates the performance of the dye removal process.

3. Both hemicelluloses derivatives (HcECA and HcEAM) were compared based on the characterization and experimental results to identify which was the better product because the results were related with the performance of dye removal application.

1.5 Thesis Outline

This report contains five chapters. The first chapter discussed the research background, problem background, objectives and scopes of this study, thesis outline and chapter summary. A critical review on this research is presented in Chapter 2. The discussions in the Chapter 2 are included the oil palm biomass, oil palm trunk, textiles wastewater treatment, batik industry, coagulation/flocculation for decolourization of textiles wastewater treatment, and hemicelluloses based flocculants from oil palm trunk (OPT). Chapter 3 outlines the research methodology which comprises research materials and experimental procedures such as synthesis and characterization. The results and discussions are presented in Chapter 4, while conclusions and recommendations of this thesis are mentioned in Chapter 5.

1.6 Summary

This study introduced an effective synthetic path for synthesizing and modification hemicellulose isolated from oil palm trunk (OPT) to produce novel hemicellulose derivatives that was expected to be the good flocculants for dye removal process. In terms of biodegradability and sustainability, flocculants derived from biopolymers could be a promising substitute to synthetic flocculants as it is considered safe and economical (Sharma *et al*., 2006). Effluent from textile industries contain multiple types of dye in which each dye different in terms of high molecular weight and complex structures. The low biodegradability of synthetic dyes was noticeable (Hsu and Chiang, 1997; Pala and Tokat, 2002; Kim *et al*., 2004; Gao *et al.*, 2007). Therefore, the use of hemicelluloses derivatives from OPT as the flocculants in dye removal process will help reduce the problem regarding on the direct discharge of dye wastewater into the sources of water like lakes, rivers and other which will pollute the water and affects the flora and fauna.

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