SYNTHESIS AND CHARACTERIZATION OF EMPTY FRUIT BUNCH CELLULOSE-BASED COAG-FLOCCULANT IN REMOVING TURBIDITY FOR WATER TREATMENT

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To my beloved parents and family for their love and support

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ABSTRACT

The increasing demand for environmentally friendly technology for drinking water treatment process has recently gained considerable attention, especially, towards application of natural-based coagulants and flocculants (coag-flocculants). Abundance of oil palm empty fruit bunch (OPEFB) generated throughout the years gives potential for this biomass to be used as a cellulose source for coag-flocculant synthesis which has so far not been studied. Thus, this study focused on the extraction of cellulose from the OPEFB using ionic liquid dissolution-alkaline treatment technique and modification of the extracted cellulose into sulphonated cellulose (s-EFBC) and quaternized cellulose (q-EFBC). The performance evaluation of cellulose-based coag-flocculant in removing turbidity of the kaolin suspension solution and river water towards drinking water treatment application was carried out using a jar test method at various experimental conditions such as the molar ratio of reactants to anhydroglucose unit of cellulose, dosage, pH, kaolin concentration and sedimentation time. The characterization results show that the physico-chemical and spectral properties of the cellulose-based coag-flocculants were greatly influenced by the modification method used. It was found that a q-EFBC9 has the highest removal of turbidity (99.11±0.11%) exceeding the alum (97.79±0.54%). Optimization results gave the overall optimum responses of interest which were turbidity removal efficiency and sludge volume index at 62.5 mg/L of coag-flocculant dosage, pH 7 and 1400 mg/L kaolin concentration. The optimum dosage was found to deliver good performance in river water, in which, encouraging results were obtained from water quality tests. The coag-flocculation kinetics was studied at various initial coag-flocculant dosages. It was found that the highest dosage of 112.5 mg/L q-EFBC resulted in the highest adsorption capacity with the highest pseudo-second order rate constant (k_2), initial adsorption rate (h) and film diffusion rate (D_f). Meanwhile, the pseudo-first order constant, shear rate and collision rate coefficient of 62.5 mg/L q-EFBC9 for flocculation kinetics were found to be the highest. The adsorption capacity of river water was lower than that of kaolin suspension at the same dosage, resulted in lower k_2, h and D_f, and similar results were found in the parameters of the flocculation kinetics study. The dewatering study showed that the coag-flocculation process with 62.5 mg/L q-EFBC resulted in the lowest specific resistance to filtration and time to filter. The OPEFB cellulose has therefore shown a good potential to be converted into coag-flocculant for drinking water treatment as well as overcoming the oil palm plantation waste disposal problems.

ABSTRAK

Peningkatan permintaan bagi teknologi mesra alam untuk proses rawatan air minuman baru-baru ini telah mendapat banyak perhatian terutamanya terhadap penggunaan pengental dan pembuku (pengental-pembuku) berasaskan bahan semulajadi. Penghasilan tandan kosong buah kelapa sawit (OPEFB) yang banyak sepanjang tahun menjadikan biojisim ini sumber selulosa yang berpotensi bagi sintesis pengental-pembuku berasaskan selulosa yang mana masih belum dikaji sehingga kini. Oleh itu, kajian ini menumpukan kepada pengekstrakan selulosa daripada OPEFB menggunakan teknik pembubaran cecair berion-rawatan alkali dan pengubahsuaian selulosa terekstrak kepada selulosa bersulfur (s-EFBC) dan selulosa berkuatenari (q-EFBC). Penilaian terhadap prestasi pengental-pembuku berasaskan selulosa dalam menyingkirkan kekeruhan ampaian kaolin dan air sungai ke arah aplikasi rawatan air minuman telah dikaji menggunakan kaedah ujian balang dengan pelbagai keadaan ujikaji seperti nisbah molar bahan tindak balas terhadap unit anhidroglukosa selulosa, dos, pH, kepekatan kaolin, dan masa pemendapan. Keputusan pencirian menunjukkan bahawa ciri-ciri fiziko-kimia dan spektra pengental-pembuku berasaskan selulosa dipengaruhi oleh kaedah pengubahsuaian yang digunakan. Didapati bahawa q-EFBC9 mempunyai penyingkiran kekeruhan tertinggi (99.10±0.11%) melebihi tawas (97.79±0.54%). Keputusan pengoptimuman menunjukkan nilai optimum untuk respon yang penting termasuk kecekapan penyingkiran kekeruhan dan indeks isipadu enapcemar adalah pada 62.5 mg/L dos pengental-pembuku, pH 7 dan kepekatan kaolin 1400 mg/L. Dos optimum itu didapati mempunyai prestasi yang baik di dalam air sungai, di mana keputusan yang memberangsangkan telah diperoleh untuk ujian kualiti air. Kinetik pengental-pembuku dikaji pada pelbagai dos awal pengental-pembuku. Didapati dos tertinggi sebanyak 112.5 mg/L q-EFBC9 menghasilkan kapasiti penjerapan tertinggi dengan pemalar kadar reaksi pseudo-peringkat kedua (k_2), kadar penjerapan awal (h), dan kadar peresapan filem (D_f) tertinggi. Manakala, pemalar pseudo-peringkat pertama, kadar ricih dan pekali kadar perlanggaran bagi 62.5 mg/L q-EFBC9 untuk kinetik pembukuan adalah yang tertinggi. Kapasiti penjerapan air sungai adalah lebih rendah daripada kaolin terampai pada dos yang sama, menghasilkan k_2, h dan D_f yang lebih rendah, dan keputusan yang sama didapati pada parameter bagi kajian kinetik pembukuan. Kajian penyahairan telah menunjukkan bahawa pengentalan-pembukuan dengan 62.5 mg/L q-EFBC9 menghasilkan rintangan spesifik penurasan dan masa penurasan paling rendah. Oleh itu, selulosa OPEFB menunjukkan potensi yang baik untuk dijadikan pengental-pembuku bagi rawatan air minuman dan seterusnya membantu dalam mengatasi masalah pembuangan sisa ladang kelapa sawit.

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ε_t	-	Turbidity removal efficiency (%)
wt.%	-	Weight percent
q_t	-	Amount of adsorbate adsorbed at time, t (mg/g)
q_e	-	Amount of adsorbate adsorbed at equilibrium (mg/g)
k_1	-	Pseudo-first-order rate constant (s^{-1})
t	-	Time (minute or second)
k_2	-	Pseudo-second order rate constant (s^{-1})
h	-	Initial rate constant of pseudo-second order (mg/g.s)
α	-	Elovich initial adsorption rate (mg/g.s)
β	-	The extent of surface coverage and activation energy for chemisorption (g/mg)
Bt	-	Boyd plot
r	-	Radius (m)
D_f	-	Film diffusion coefficient (cm^2/s)
ξ	-	Zeta potential (mV)
X_i	-	Real value
x_i	-	Coded value
Xo	-	Value at the centre point
δX	-	Step change
Y	-	Predicted response
b_i	-	Linear coefficient
b_{ii}	-	Quadratic coefficient
b_{ij}	-	Interaction coefficient
R^2	-	Coefficient of determination
R^2_{adj}	-	Adjusted coefficient of determination
C_o	-	Initial concentration of coag-flocculant at t = 0 (mg/L)

C_t	-	Residual concentration of coag-flocculant in the kaolin suspension at time t (mg/L)
V	-	Volume (mL or L)
m	-	Mass of kaolin (mg)
N_T	-	Number of concentration at time *t* (particle/m^3)
No	-	Number of concentration at t = 0 (particle/m^3)
K	-	Flocculation pseudo-first order rate constant (s^{-1})
ρ_k	-	Density of the particle (kg/m^3)
b	-	A slope determined from the plot of t/vol vs vol (m^6/s)
P	-	Pressure of filtration (N/m^2)
A	-	Area of filtration (m^2)
μ	-	Filtrate viscosity (Ns/m^2)
w	-	Dry mass of cake solids deposited per volume of filtrate (kg/m^3)
N	-	Normality
A	-	Absorbance at 205 nm
W	-	Oven-dry mass (mg or g)
df	-	Dilution factor
l	-	Cell path length (1 cm)
a	-	Absorptivity equal to 110 L/g.cm
W_o	-	Initial mass before oven-dry (g)
W_1	-	Weight of ash (g)
W_2	-	Weight of ambient-dry OPEFB (g)
M_w	-	Weight-average molecular weight (g/mol)
M_n	-	Number-average molecular weight (g/mol)
DS_N	-	Subtitution degree of nitrogen
DS_S	-	Substitution degree of sulfur
$\%N$	-	Amount of nitrogen content detected by elemental analyzer (wt.%)
$\%S$	-	Amount of sulfur content detected by elemental analyzer (%)
$T_{initial}$	-	Initial turbidity (NTU)
$T_{treated}$	-	Final turbidity (NTU)
D_1	-	Initial DO of sample (mg/L)
D_2	-	Final DO of sample (mg/L)

B_1	-	Initial DO of seed (mg/L)
B_2	-	Final DO of seed (mg/L)
w_{RF}	-	Weight of residue and filter paper (g)
w_F	-	Weight of filter paper (g)
I_{AM}	-	Height of the minimum XRD spectrum
I_{002}	-	Height of the 002 peak XRD spectrum
λ	-	Wavenumber (cm^{-1})
T	-	Transmittance (%)
Std. Err.	-	Standard error
q_{exp}	-	Experimental equilibrium sorption capacity (mg/g)
q_{theory}	-	Theoretical equilibrium sorption capacity (mg/g)
q_{cal}	-	Calculated sorption capacity based on theoretical equation (mg/g)
ARE	-	Average relative error
MPSD	-	Marquardt's percent standard deviation
ø	-	Volume fraction
G	-	Shear rate (s^{-1})
k	-	Orthokinetic collisions rate coefficient (m^3/s)
k_{ij}	-	Orthokinetic collisions rate coefficient of i- and j-particle (m^3/s)
J_{ij}	-	Number of collisions of i- and j-particles (s^{-1})
C_k	-	Kaolin concentration (mg/L)

LIST OF ABBREVIATIONS

[bmim][Cl]	-	1-butyl-3-methyl-imidazolium chloride
^{1}HNMR	-	Hydrogen nuclear magnetic resonance
AGU	-	Anhydroglucose unit
$AlCl_3$	-	Aluminium chloride
$AlK(SO_4)_{2.}12H_2O$	-	Aluminium potassium sulphate dodecahydrate
ANOVA	-	Analysis of variance
ASTM	-	American standard test method
BOD_5	-	Biological oxygen demand
CCD	-	Central composite design
CH_4	-	Methane
CHPTAC	-	3-chloro-2-hydroxypropyltrimethylammonium chloride
CNT	-	Carbon nanotube
CO	-	Carbon monoxide
Coag-flocculant	-	Coagulant and flocculant
COD	-	Chemical oxygen demand
CrI	-	Crystallinity index
DI	-	Double distilled
DMSO-d_6	-	Deurated dimethyl sulphoxide
DO	-	Dissolved oxygen
DOC	-	Dissolved organic carbon
DS	-	Degree of substitution
DTG	-	Derivative weight
EDX	-	Energy-dispersive x-ray
FESEM	-	Field emission scanning electron microscopy
F-test	-	Fisher test
FTIR	-	Fourier transform infrared
εGPC	-	Gel permeation chromatography

H_2SO_4	-	Sulphuric acid
HNO_3	-	Nitric acid
HPLC	-	High performance liquid chromatography
IL	-	Ionic liquid
KIO_4	-	Sodium metaperiodate
KOH	-	Potassium hydroxide
MS	-	Mean square
$NaClO_2$	-	Sodium chlorite
$NaHSO_3$	-	Sodium bisulphite
NaOH	-	Sodium hydroxide
NOM	-	Natural organic matter
NTU	-	Nephelometric turbidity units
OPEFB	-	Oil palm empty fruit bunch
PACl	-	Polyaluminium chloride
PAM	-	Polyacrylamide
PDADMAC	-	Polydiallyldimethyl ammonium chloride
PDADMAN	-	Polydiallyldimethyl ammonium nitrate
PDADMAS	-	Polydiallyldimethyl ammonium sulphate
p-value	-	Probability value
q-EFBC	-	Quaternized cellulose
RSM	-	Response surface methodology
s-EFBC	-	Sulphonated cellulose
SEM	-	Scanning electron microscopy
SRF	-	Specific resistance of filtration
SVI	-	Sludge volume index
TGA	-	Thermogravimetric analysis
THF	-	Tetrahydrofuran
TSS	-	Total suspended solid
TTF	-	Time to filter
XRD	-	X-ray diffraction

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Cellulose is one of the most abundant and naturally occurring biopolymer which is commonly found in the cell wall of plants and certain algae. It has long been investigated as a new green source to replace non-renewable materials and chemicals. The common sources of cellulose are from wood and non-woody plants. The use of non-woody plant waste residues such as rice and pineapple residues, coconut husks and oil palm biomass which are available in large quantities and have unexplored potential as cellulose raw materials have attracted much attention recently. In 2010, Malaysia alone has produced about 80 million tonnes of dry solid oil palm biomass comprising of oil palm empty fruit bunches (OPEFB), fronds, trunks, palm kernel shells, and mesocarp fibres from plantation and palm oil processing activities. This quantity is expected to increase from 70 to 110 million tonnes of dry solids by 2020 (Agensi Inovasi Malaysia, 2013). Thus, it is interesting to explore the production of cellulose from this inexpensive lignocelluloses source which could be further developed as value-added products.

As an important class cellulosic materials, cellulose derivatives have very wide applications in industries concerned with oilfield treatments, medical products, protective colloids, coating, surfactants, hair conditioners, antistatic agents, dispersion agents, adhesives and textiles (Dumitru, 2002; Hon, 1996). Another potential application of cellulose derivatives is as a coag-flocculant in water treatment processes. Coag-flocculation is a regular technique to remove organic particles from water in conventional water treatment processes by inducing destabilization and agglomeration of dispersed colloidal particles into larger aggregates or flocs. Basically, the coagulation process involves the dissolution of coag-flocculants, adsorption of coag-flocculants onto particle surface, and reconformation of the coag-flocculants. The flocculation process involves agglomeration of particles which mutually attached by the hydraulic shear force in the presence of coag-flocculation agents or the entrapment caused by longer molecular chains of the agent used. One of the most widely used material is inorganic salt aluminium sulphate ($Al_2(SO_4)_3$), also known as alum. Alum has several advantages such as good performance as well as low cost, easy to use and easy to obtain. Besides alum, other inorganic salts such as ferric chloride, polyaluminium chloride and synthetic coagulants of polymer-based organic such as polyacrylamide have been used in practice.

Surface waters such as river water are sources of drinking water that contain impurities harmful to the human body. For example, the existence of humic substances badly affects drinking water quality since they cause aesthetic problems such as unpleasant colour, odour, and taste. These substances also act as precursors for the formation of chlorinated compounds, facilitate substrates for bacterial growth, carriers of adsorbed hydrophobic organic and inorganic chemicals, add to the coag-flocculants amount and increase the demand for disinfectant in water treatments. They also have complex properties which include association with toxic elements and micro-pollutants (Exall and vanLoon, 2000). Thus, the treatment of source waters prior to being consumed as drinking water is compulsory and should meet the regulations and limitations stated by the authorities to ensure its safety and cleanliness.

Recently, there are increasing demands for developing environmental friendly and natural-based coag-flocculants to replace the synthetic ones. Thus, nowadays, attention is being shifted to the development of new classes of non-toxic, biodegradable, readily available and economical alternatives. In this regard, an increasing number of studies focusing on natural products have been conducted. For example, modifications on tamarind kernels (Pal *et al*., 2012), palm rachis (Khiari, 2010), *Moringa oleifera*, *Opuntia ficus-indica* (Beltrán-Heredia and Sánchez-Martín, 2009) and guar gum (Tripathy *et al*., 2008) as materials to be used as coag-flocculants. In comparison with synthetic polymers, natural coag-flocculants are safe for humans, biodegradable, have a wider effective dosage range for coag-flocculation of colloidal suspensions and do not produce secondary pollution (Sanghi *et al*., 2006). Therefore, the naturally occurring polymer from plantation waste, namely OPEFB, is interesting to be investigated as a coag-flocculant for the removal of turbidity in drinking water treatment processes.

1.2 Problem Statements

As one of the largest palm oil producers and exporters in the world, Malaysia generates huge amounts of solid wastes from its plantation and milling activities. It was reported that approximately 90% of OPB was produced from oil palm trees involved in the both activities (Basiron, 2007). Dealing with such quantity of waste is a gigantic task, as if not tackled properly may lead to environment degradation (Singh *et al*., 2010). This biomass is rich with useful materials known as lignocelluloses compounds. Unfortunately, nowadays, most of them are either burnt in the open or disposed off in waste ponds, though. Therefore, the exploitation of oil palm biomass should be maximized to add sustainability and profit on the relevant industry (Abdul Khalil *et al*., 2012).

One of the major compounds in OPB is cellulose. This natural polymer has versatile uses in diverse applications when derived. Coag-flocculation processes are one of the potential applications for cellulose derivatives. This application faces problems due to the fact that the commercially available coag-flocculant, alum has gained considerable concern due to its toxicity as a high concentration of these materials in water is likely to implicate human health. To minimize or substitute the usage of alum, synthetic organic polymer such as polydiallyldimethyl ammonium (Tian *et al*., 2006), poly(acrylamide-co-acrylic acid) (Liu *et al*., 2000), epichlorohydrin–dimethylamine (Wang *et al*., 2012), etc., have been introduced. The advantages of the polymeric coag-flocculants are they require lower dosages and producing denser sludge. However, the cost is also increasing due to the increasing price of their non-renewable raw materials originated from petroleum.

In line with the increasing demand for healthy, environmental friendly and cheap technologies, natural polymer-based coag-flocculants including cellulose have received significant attention as an alternative to the inorganic and synthetic polymer coag-flocculants (Lu *et al*., 2011). Recently, the utilization of water soluble cellulose derivatives as coag-flocculants has been reported. Cellulose has been grafted with acrylamide (Song *et al*., 2011), cationized with quaternary ammonium groups (Song *et al*., 2010; Liimatainen *et al*., 2011; Hebeish *et al*., 2010) and Girards's reagent (Sirviö *et al*., 2011), and graft copolymerized with polyacrylamide and vinyl sulphonic group (Biswal and Singh, 2006; Sand *et al*., 2010), as attempts to improve its properties as coagulant or flocculant. Therefore, it is interesting to explore the potential of cellulose extracted from OPB, as coag-flocculant. In this study, OPEFB was chosen as the raw material since it contains high cellulose content.

1.3 Objectives of the Studies

Based on the identified problem statements, this study embarks on the following objectives:

i. To isolate, characterize and modify cellulose extracted from OPEFB as coag-flocculants.

ii. To study the performance of the cellulose-based coag-flocculants in a synthetic kaolin suspension and optimize the performance of the selected coag-flocculants, followed by the performance evaluation of the selected coag-flocculants on river water for drinking water usage.

iii. To study the coagulation and flocculation kinetics as well as sludge dewatering property of the selected cellulose-based coag-flocculation processes.

1.4 Scopes of the Studies

In this study, the cellulose extracted from OPEFB was modified to be used as coag-flocculants for treatment of drinking water. The extraction was carried out using a dual-technique involving dissolution in ionic liquid (IL) and treatment with NaOH solution. The physico-chemical properties of the cellulose extracted from OPEFB were characterized using a Fourier transform infrared (FTIR) to determine the functional groups, gel permeation chromatography (GPC) to determine the average molecular weight, and thermogravimetric analysis (TGA) to study its thermal degradation. The modifications were done using sulphonation and quaternization processes and the molar ratios of the active reagents used in the preparation were varied. The cellulose-based coag-flocculants were characterized using FTIR and hydrogen nuclear magnetic resonance (^{1}HNMR) to confirm the sulphonation as well as quaternization reactions on the cellulose, and a CHNS elemental analysis was used to determine the degree of substitution (DS).

The performance of the cellulose-based coag-flocculants was evaluated using a jar test method by manipulating one parameter while fixing other parameters. The manipulated parameters were the coag-flocculants' molar ratio (reagent:AGU), dosage, the pH of synthetic kaolin solution, sedimentation time, and kaolin concentration. The performance of the coag-flocculants was denoted by turbidity removal efficiency, ε_t (%) which was determined using a turbidimeter and compared with the commercial alum. The coag-flocculant with the highest efficiency was selected for further studies. The optimization experiment was conducted by choosing a coag-flocculant with the best performance and evaluated using the response surface methodology (RSM). The independent parameters were coag-flocculants dosage, pH of the synthetic kaolin solution and sedimentation time, while the dependent parameters were turbidity removal efficiency and sludge volume index

(SVI). River water was used to investigate the performance of the selected coag-flocculant for drinking water treatment purposes. Several water quality tests were conducted which include turbidity, total suspended solid (TSS), biochemical oxygen demand (BOD_5), chemical oxygen demand (COD), colour determination, total solid content, SVI and heavy metals determination.

In order to study the kinetics of the coag-flocculation process, several analyses were conducted. A high performance liquid chromatography (HPLC) analysis was used to study the adsorption kinetics of the coagulation process which had involved several existing kinetic models (i.e. pseudo-first order, pseudo-second order, Elovich and Fick's film diffusion). The flocculation kinetics experiment was done using a gravimetrical technique and the orthokinetic theory was used to describe the kinetic process. To determine the sludge property, a sludge dewatering experiment was conducted using a Buchner funnel test and the specific resistance of filtration (SRF) and time to filter (TTF) were determined. In this study, two types of suspension were evaluated, namely the kaolin suspension and river water.

1.5 Thesis Outline

This report is organized into five chapters. Chapter 1 presents general information regarding the background, problem statement, objectives and scopes of the studies. Chapter 2 generally discusses the subject of coag-flocculation in drinking water treatment processes. The first section of the chapter deliberates about the sources of cellulose, cellulose isolation and modifications, as well as cellulose applications as a coag-flocculant for various purposes. Then, the second section discusses on coag-flocculation processes, followed by a third section which discusses on coag-flocculation fundamentals, wherein the effect of parameters, mechanisms as well as the kinetics of the coag-flocculation and dewatering processes were included.

Chapter 3 discusses on the methodologies used in order to achieve the objectives. This chapter comprises of materials used, the experimental procedures, and the analytical characterization procedures. The experimental procedures include extraction and modification methods, coag-flocculation experiments, optimization, kinetics and dewatering studies. The characterization section justifies the procedures used to characterize the coag-flocculants proximately and ultimately. The analytical procedures describe the methods and calculations used for water quality tests of the river water. Chapter 4 presents the results and discussions to meet the objectives of the study. This includes investigation on the extraction and modification of cellulose-based coag-flocculants, their performance in removing turbidity of kaolin suspension, the response optimization of selected coag-flocculants and its potential in removing the turbidity of river water, the coagulation and flocculation kinetics processes, and sludge dewatering property. Finally, Chapter 5 gives a summary for the findings of the study and recommendations for future work.

1.6 Summary

The exploitation of OPEFB as a raw material for cellulose isolation is interesting in terms of environmental and economic value. Besides converting this abundant biomass into value added products, the modification of cellulose into biodegradable coag-flocculants offer safer drinking water processing as the commercially available coag-flocculants are harmful to humans and can cause mechanical-related problems. Thus, it is interesting to study the performance of cellulose-based coag-flocculants extracted from OPEFB as substitutes to synthetic coag-flocculants. Two types of cellulose-based coag-flocculants were fabricated and characterized in this study and the performances of both samples were compared with alum. This experiment was also considered as a preliminary step to select the coag-flocculants’ important parameters as well as to determine the range of the parameters for further study in the optimization experiment. Further analyses involving the optimization of the coag-flocculation process provides information regarding the best conditions for the highest performance of coag-flocculant which was later used in the river water treatment analysis. The kinetics experiment elucidated the possible adsorption and aggregation occurring during the coag-flocculation processes. Finally, the sludge property in terms of dewatering was also evaluated.