UNIVERSITI PUTRA MALAYSIA

TREATMENT OF WASTEWATER FROM KENAF WATER-RETTING PROCESS

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By

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Treatment wastewater from kenaf-retting process was implemented using three types of treatment process, namely coagulation, sequencing batch reactor (SBR) and adsorption process. Each of the tested process has advantages and disadvantages. Characteristic of wastewater was measured to determine presence of chemical composition. Concentration of chemical oxygen demand and colour were recorded in high concentration, which is more than 2000mg/L and 1000mg/L respectively. Most of parameters tested did not comply with the standard and regulation that regulated by Department of Environment (DOE) Malaysia.

In coagulation process, five types of coagulants were selected based on its reputation achieved in previous research. There are ferric chloride, aluminium sulphate, ferric sulphate, *Moringa oleifera* and *Jatropha curcas*. Results obtained from the experiment have given satisfactory results; more than 90% of turbidity was removed from the wastewater by all coagulants tested. All coagulants tested except ferric sulphate required acidic condition to work at optimum condition. Nevertheless, for the removal of COD all coagulants showed an average performance.

Then, the wastewater was tested with biological treatment, which activated sludge from nearby sewerage treatment plant used as source of bacteria. Diversity of microorganisms live in the sludge is a main criterion for the selection. Acclimatization process was implemented before biological treatment executed. Achromobacter, bacillus and acinetobacter were identified as dominant species lived in the wastewater. For biological treatment, sequencing batch reactor was selected to treat the wastewater. Two main parameters were tested; there are food-to-microorganisms (F/M) ratio and hydraulic retention time (HRT). SBR shows the best condition at low F/M ratio, which is 0.25, and the lowest of COD concentration recorded was 163mg/L. whilst, for HRT, SBR showed the best efficiency at HRT 24 per cycle. At higher HRT, the performance of SBR becomes less efficient. The lowest COD value in the final effluent for HRT24 was recorded at 114mg/L.

The last treatment process tested was adsorption process. Activated charcoal was selected as an adsorbent. The highest uptake rate in various initial pH was found at pH8, and the adsorption process efficiency in this experiment was pH dependent. The increasing amount of adsorbent dosage enhanced the COD removal rate, but not the sorption capacity. However, 1.0g of activated charcoal was selected as the best dosage when the cost of adsorbent and treatment take into consideration and the value of COD in the effluent was 339mg/L. In equilibrium studies, COD concentration had shown well agreed with the Redlich-Peterson with high correlation coefficient values compared to other models

Combination of coagulation process with SBR and activated charcoal showed a good performance and high efficiency in removing total suspended solids, chemical oxygen demand and colour. Coagulation process is capable to remove COD and TSS averagely around 80% and 40% respectively. Through SBR-AC process, the value of COD become lower with the reduction recorded around 133mg/L only. Overall the integration process was the best treatment process compared to other three processes.

Abstrak tesis yang dikemukakan kepada Senat Unversiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

RAWATAN AIR SISA DARI PROSES RENDAMAN KENAF

Oleh

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Rawatan air sisa dari proses pemisahan fiber Kenaf dilakukan melalui empat jenis rawatan iaitu process pengumpalan, reaktor penjujukan berkelompok, process penjerapan dan proses pengumpalan bersama reaktor penjujukan berkelompok yang berintegrasi dengan bahan penjerap. Setiap proses yang diuji memberikan kelebihan dan kekurangan yang tersendiri. Karakter bagi air sisa diuji terlebih dahulu bagi memastikan komposisi kimia yang hadir didalamnya. Kehadiran keperluan oksigen kimia, pepejal dan warna yang tinggi melebihi 1000mg/L. Kebanyakan parameter yang diuji menunjukkan bahawa kandungan didalam air sisa tersebut tidak mematuhi piawaian yang ditetapkan oleh Jabatan Alam Sekitar Malaysia.Kandungan bahan organik dan bukan organik juga diuji bagi mengetahui bahan spesifik yang terkandung didalam air sisa yang dikaji. Elemen bukan organik yang diuji seperti mempunyai kepekatan yang rendah. Manakala bahan organik pula terdiri daripada bahan semulajadi yang terdapat pada tumbuhan kenaf.

Melalui proses pengumpalan yang dilakukan, lima jenis pengumpal telah dipilih berdasarkan reputasi yang dicapai melalui kajian yang lepas. Ia adalah, *ferric chloride, aluminium sulphate, ferric sulphate, Moringa Oleifera* dan *Jatropha Curcas*. Melalui eksperimen yang telah dijalankan, didapati bahawa proses tersebut menunjukkan hasil yang sangat memuaskan dimana, penyingkiran kekeruhan dari sampel air sisa melebihi 90% dari jumlah keseluruhan kepekatan kekeruhan dari sampel. Kesemua pengumpal kecuali *ferric sulphate* memerlukan keadaan berasid bagi membolehkannya berfungsi secara optima. Namun begitu, dari segi penyingkiran keperluan oksigen kimia berada diparas sederhana bagi kesemua jenis pengumpal yang diuji.

Seterusnya, air sisa diuji pula dengan rawatan secara biologi dengan menggunakan bakteria yang diambil dari loji rawatan kumbahan yang berdekatan. Melalui proses penyesuaian yang dijalankan, terbukti bakteria yang digunakan mampu merawat air sisa dengan baik. *Achromobacter, bacillius* dan *acinetobacter* merupakan spesis yang mendominasi didalam sampel air sisa . Bagi ujian kaedah penjujukan berkelompok, dua jenis parameter yang diuji iaitu nisbah *food-to-microorganisms*

(F/M) dan juga masa tertahan hidraulik (HRT). Melalui ujian yang telah dijalankan, didapati bahawa, semakin rendah kadar nisbah F/M yang digunakan semakin bagus reaktor merawat air sisa. Nisbah F/M yang paling berkesan dalam rawatan ini adalah 0.25 dan nilai COD didalam sampel akhir adalah sekitar163mg/L. Manakala bagi masa tertahan hidarulik pula yang diperlukan oleh reaktor adalah lebih kurang 24 jam bagi satu pusingan. Semakin tinggi masa tertahan hidraulik yang digunakan semakin rendah kecekapan reaktor merawat air sisa yang diuji. Nilai COD paling rendah yang diperolehi adalah sekitar 114mg/L sahaja.

Seterusnya, proses penjerapan dengan mengunakan bahan karbon teraktif sebagai penjerap. Berdasarkan daripada kepeutusan yang diperolehi menunjukkan bahawa bahan penjerap berupaya menyingkirkan kepekatan COD pada keadaan pH8 dan 283mg/L merupakan nilai COD akhir yang dicatatkan. Bagi ujian perbezaan berat bahan karbon teraktif yang dijalankan menunjukkan bahawa semakin berat bahan karbon teraktif yang digunakan semakin tinggi kadar penyingkiran kepekatan COD diperolehi. Namun begitu, dengan mengambila kira kos bahan dan rawatan, 1.0g bahan penjerap dikira yang paling baik dalam menyingkirkan bahan enapcemar daripada air sisa. Nilai COD akhir yang dicatatkan adalah 339mg/L.

Rawatan melalui proses pengumpalan bersama kaedah penjujukan berkelompok yang berintegrasi dengan bahan penjerap (SBR-AC) merupakan kaedah yang keempat yang diuji. Melalui ujian yang dijalankan, didapati bahawa, kombinasi kaedah rawatan ini mempunyai kecekapan yang tinggi dan efisien dalam menyingkirkan permintaan oksigen kimia, pepejal terampai dan warna yang menjadi punca utama kepada masalah ini. Proses penjerapan berjaya menurunkan kadar kepekatan COD dan pepejal terampai masing-masing sekitar 80% dan 40%. Seterusnya air sisa itu dirawat dengan kaedah SBR-AC, dan nilai COD menurun sehingga 133mg/L sahaja, manakala TSS pula mengalami peningkatan sedikit. Secara keseluruhannya, keputusan ujian yang telah dijalankan menunjukkan bahawa proses integrasi ini berjaya menurunkan kadar kepekatan bahan enapcemar dengan begitu baik sekali. Secara keseluruhannya, proses integrasi menunjukkan prestasi yang terbaik berbanding dengan kaedah rawatan yang lain.

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LIST OF ABBREVIATIONS

Notations / symbols		
°C	Celcius	
Å	Length, = 10^{-10} m	
AA	Activated alumina	
ADMI	American Dye Manufacturer Institute	
AlS	Aluminium sulphate	
APHA	American Public Health Association	
AS	Acitivated sludge	
B.C	Before century	
b_j	Redlich-Peterson constant	
BOD	biological oxygen demand	$mg.L^{-1}$
BOD5	biological oxygen demand for 5-days	
Cd	Cadmium	
C_e	Bulk phase concentration	$mg.L^{-1}$
C_o	Initial concentration	$mg.L^{-1}$
CO	Carbon monoxide	
Co	Cobalt	
CO2	Carbon Dioxide	
COD	chemical oxygen demand	$mg.L^{-1}$
Cr	Chromium	
C_t	Concentration of the solution at time t	$mg.L^{-1}$
Cu	Copper	
DOE	Department of Environment	
F/M ratio	food-microorganism ratio	
FC	Ferric chloride	
FS	Ferric sulphate	
g	gram	
GAC	Granular activated carbon	
GCMS	Gas Chromatography Mass Spectrophotometer	
HCl	Hydrochloric acid	
hr	hour	
HRT	hydraulic retention time	hr
ICP	Inter couple Plasma	
IUPAC	International Union of Pure and Applied Chemistry	
K_F	Freundlich adsorption capacity	mg/g
kg/mol	kilogram per mol	
K_j	Adsorption capacity	$L.g^{-1}$
K_L	Langmuir constant	$L.g^{-1}$

L	litre	
l/g	litre per gram	
l/mg	litre per miligram	
m	Adsorbent mass	g
mg	miligram	
mg/L	miligram per litre	
min	minute	
min	minute	
mL	mililitre	
MLSS	Mixed liqour suspended solids	$mg.L^{-1}$
n	Surface heterogeneity	
N	normality	
NA	Not available	
NaOH	Sodium Hydroxide	
NTU	Nephelometric Turbidity Unit	
O_2	Oxygen	
PAC	powdered activated carbon	
PtCo	Platinum Cobalt	
q_e	Solid phase concentration at equilibrium	mg/g
$q_{e,calc}$	Predicted solid phase concentration at equilibrium	mg/g
$q_{e,exp}$	Solid phase concentration at equilibrium obtained from experiment	mg/g
q_t	Solid phase concentration at time t	mg/g
R_L	Dimensionless separation factor	
rpm	rotation per minute	
SBR	Sequencing batch reactor	
SS	Suspended solids	
t	Time	minute
TDS	Total dissolved solids	$mg.L^{-1}$
TSS	Total suspended solids	$mg.L^{-1}$
V	Volume of solution	mL
VSS	volatile suspended solids	$mg.L^{-1}$
α_L	Energy of adsorption	$dm^3.mg^{-1}$
β	Heterogeneity factor	

CHAPTER 1

INTRODUCTION

1.1 Background of studies

Recently, a lot of researchers focused on natural fibres in finding new materials to produce environmental friendly and renewable products. There are numbers of researches have been done on various types of natural fibres such as jute, sisal, hemp and kenaf due to capability of these plants to meet the requirement as eco-friendly products. Among natural fibres, kenaf has its own reputation as an alternative material in industry and research field. Kenaf has been recognized as one of the most important fibres in product development like composites, pulp/paper, particle boards etc.

Bast-fibres that found in the kenaf plant are playing an important role as a raw material for plant-based or bio-composite products. Nevertheless, bast-fibres cannot be used directly as a raw material because it needs to be separated into individual fibre. This individual fibre can be obtained through retting process.

Retting process can be defined as a slow degradation process that can take up a few days to complete. There are various types of retting, for an example using chemical, dew- or water-retting. Commonly water-retting procedure is selected because it is simple and often produced a good quality of fibre. However, large amount of water in fibre separation (retting) process is required to obtain the fibres. Typically water that has been used in the process will be released to the watercourse without any treatments. The condition of wastewater that turbid and produces odour makes it unacceptable by public. This problem can be resolved with an appropriate treatment process.

For that reason in this research study, wastewater is characterized and the treatment process was designed to treat the wastewater. Therefore, coagulation process, sequencing batch reactor (SBR) and adsorption process were proposed to treat the wastewater. These processes are well-known for their effectiveness in treating wastewater either from municipal or industrial.

1.2 Problem statement

i. Generation of wastewater from kenaf water-retting process

At present, industrial sector are producing fibres in large quantity through water-retting process, hence, the process generating enormous amount of wastewater from the separation process. Generation of wastewater from kenaf retting process in Malaysia is unknown due to lack of information. Nevertheless, according to Mondal & Kaviraj (2008) as reported by Huda et al. (2012) for 10,000kg of jute, approximately 432m^3 of water is used in water-retting process. Since kenaf and jute are from one family known as *Malvaceae* so the retting process is similar. In the future, amount of wastewater will be increased tremendously when the amount of fibre production will be greater than before as kenaf become one of the commodities in Malaysia. An effective wastewater treatment process is necessitated to protect the environment, to fulfil the standard required by authority and also to help to recover water resource. However, cost of operation and land area might be the main obstacles for small industry and farmers to treat the wastewater properly.

ii. Presence of foreign matter and compound from the water-retting process to aquatic environment

Solids either suspended or dissolved can be found due to presence of fibres and other component during the retting process. These solids have caused the wastewater become cloudy. Organic substances also can be related to increasing turbidity in the wastewater. A material such as lignin was diffused into the water during the retting process. Therefore, high turbidity which consists of suspended particles, colloids and substances in the wastewater will induce interfere an aquatic ecosystem when the wastewater release into the ecosystem without any treatment due its potential to deplete oxygen (O_2) in the water.

The existence of colour is often related to the presence of certain substance in the wastewater. Generally, colour in the wastewater is unacceptable by the public. Unlike man-made coloured wastewater, it is quite difficult to get rid of the colour from the wastewater without knowing type of compound that responsible for its existence. Furthermore, if the wastewater is released at the point for water intake, it will affect quality of water which will increase the cost of water treatment.

iii. Reclaim and reuse treated wastewater for irrigation

Clean water is an important resource for human and become main attention recently due to declination of water source around the world. The reduction source occurred mainly due to water pollution caused by human activities. Therefore, recycle and reuse program was introduced to optimize an application of treated wastewater for other application such as watering public park, field or farm. However, reuse the wastewater for farming application it is depend on the origin of wastewater, quality of effluent and also whether it is for edible crops or not.

1.3 Objective of studies

The detailed research objectives are listed below.

i. to evaluate the performance of various coagulants in coagulation/flocculation process in treating kenaf-retting wastewater

ii. to evaluate the performance of biological treatment, Sequencing Batch Reactor (SBR) in treating kenaf-retting wastewater

iii. to evaluate the performance of adsorption process in the treatment of kenaf-retting wastewater.

iv. to evaluate performance of integrated treatment process by combining coagulation, SBR and adsorption process as one unit treatment to treat kenaf-retting wastewater

1.4 Scope of studies

There are various types of treatment process consist of physical, chemical and biological were selected and investigated in this study; coagulation, adsorption and sequencing batch reactor (SBR) process and also combination of these three methods in as one treatment unit. These processes are well-known for its good performance in treating municipal or industrial wastewater. The effluent was also investigated for its potential to reuse and reclaim for other activities. The scopes can be summarized as follows;

i. Analyze the characteristic and behaviour of wastewater

There were few or none previous study on characteristic of kenaf-retting wastewater has been reported. For that reason, characteristic and behaviour study of this wastewater is important before any treatment can be done. Therefore, several analyses were implemented to fulfil the intent. American Public Health Association (APHA) standard procedure has been used to serve as guideline to analyze the kenaf-retting wastewater.

ii. Evaluate performance of selected coagulant and its optimum condition to treat the wastewater

Due to existence of suspended solids (SS) and colloids in the wastewater, thus coagulation process was selected to treat the wastewater. Coagulation is managed to remove any particulate and substances in the wastewater by using several types of selected coagulants that consists of chemical and natural coagulants. Optimum condition for every type of coagulant was determined in terms of coagulant dosage and pH of wastewater sample.

iii. Acclimatization of activated sludge from other source in the wastewater

Activated sludge (AS) from sewerage treatment plant was used as a seeding for SBR since it is difficult to find microbes in the wastewater. Diversity of microorganisms live in the sludge makes it easy to receive different kinds of substrate and pollutants. Acclimatization process was run to introduce the AS with the new wastewater and performance of microorganisms was monitored. Batch method was took place in the acclimatization process, which wastewater sample was introduced to AS for certain period of time and then withdrawn the sample. After that the process was continue introduced new samples until the AS reaches its stabilization. Acclimatized sludge was cultivated and isolated for microbe's species identification. After the AS was acclimatized with the wastewater, the AS is ready to be used as inoculums in biological treatment process.

iv. to examine the effectiveness of sequencing batch reactor (SBR)

Wastewater produced from water-retting process contains high organic compound and unpleasant odour. Biological treatment like sequencing batch reactor (SBR) has capability to treat the wastewater with a lot of advantage such as capability to receive high shock of loading rate and eliminate odour. Acclimatized AS was used as inoculums in the SBR process. Main parameters were exploited in the study such as hydraulic retention time (HRT) and food to microorganism (F/M) ratio.

v. to explore the potential and the efficiency of adsorption process

Adsorption process was considered in this study due to its efficiencies in treating innumerable types of wastewater. Granular charcoal activated carbon was picked as an adsorbent in this study. Batch experiment was implemented in the process such as equilibrium study, effect of adsorbent dosage and pH of sample.

vi. to measure performance and effectiveness of integrated system to treat the wastewater

Coagulation process as a pre-treatment process, combining with SBR that integrated with activated carbon was implemented in this study. Optimum condition employed in the experiment was obtained from previous experiment – coagulation, SBR and adsorption.

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