CHITOSAN-FILLED FILTER MEDIA FROM LIGNOCELULOSIC BIOMASS

AZIATUL NIZA SADIKIN

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DEDICATION

This thesis is especially dedicated to my husband, Ahmad Nizar Mohamad and my beautiful, beloved children, Ahmad Aiman, Aisyah Najla and Ameena, as well as my beloved parents. You are all the sources of my strength. May Allah keep us all together, Amiin.

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ABSTRACT

The aim of this research is to evaluate the feasibility of the fibrous media for removal of total suspended solid and oil grease from palm oil mill effluent (POME). The first part of the research deals with the fabrication and characterization of the non-woven filter media. Wet lay-up method was adopted for filter fabrication where empty fruit bunches (EFB) were matted together with chitosan polymer in non-woven manner. The percentage of water sorption is significantly lower for chitosan-filled filter media as compared to binder-less filter media and the percentage decreases as the chitosan concentration increases. The increase of tensile strength of the chitosan-filled filter media could be attributed to the presence of the hydrogen bond between cellulose fibre and chitosan molecules as indicated in Fourier transform infrared spectroscopy spectra and good dispersion of chitosan solution onto the surface of filter media as suggested by the optical images. The Taguchi method revealed that the amount of chitosan loading had the most significant effect on filter media permeability compared to chitosan concentration, filter thickness, fabrication method and the fibre size. The filter with alkali-treated fibre showed a tighter network structure compared to the filter prepared from untreated EFB. The second part of the research studied the performance of the newly developed non-woven filter media. Filtration flow-rate, filter depth, amount of chitosan loading in fibres and influent concentration were studied using the Response Surface Methodology. Filtration results indicated that chitosan-filled filter media filtration only removed up to 28.14% of total suspended solid, 29.86% of oil and grease, and 8.13% of chemical oxygen demand. Chitosan loading in the fibres was the most significant factor affecting the treatment of POME by chitosan-filled filter media filtration. The filter with a lower depth was able to remove particulates quickly but due to its thinness could not remove substantial quantities of the particulates over a long period of time. POME with a higher influent concentration led to a more rapid pressure drop during filtration process. In addition, all filter media suffered permeability loss and were easily clogged, which renders it unusable for long term filtration.

ABSTRAK

Tujuan kajian ini adalah untuk menilai kemungkinan untuk media bergentian menyingkirkan pepejal terampai dan minyak gris daripada sisa kilang kelapa sawit (POME). Bahagian pertama dalam penyelidikan ini berkenaan dengan fabrikasi dan pencirian media penapis bukan tenunan. Media penapis difabrikasi dengan mengunakan kaedah bengkalai basah di mana tandan buah kosong (EFB) dicampur bersama dengan polimer kitosan. Peratusan penyerapan air adalah jauh lebih rendah untuk penapis yang mempunyai pengikat berbanding dengan media penapis tanpa pengikat dan peratusan penjerapan semakin menurun dengan pertambahan kepekatan kitosan. Peningkatan kekuatan media penapis kitosan boleh dikaitkan dengan kehadiran ikatan hidrogen antara gentian selulosa dan molekul kitosan seperti yang ditunjukkan oleh analisis spektroskopi inframerah transformasi Fourier dan taburan kitosan yang sekata di atas permukaan media penapis seperti yang ditunjukkan oleh imej optik. Kaedah Taguchi menunjukkan bahawa jumlah kitosan di dalam penuras merupakan kesan yang paling ketara terhadap kebolehterapan penuras berbanding dengan kepekatan kitosan, ketebalan penapis, kaedah fabrikasi dan saiz gentian. Penapis dengan serat yang dirawat oleh pelarut alkali menunjukkan struktur rangkaian yang lebih ketat berbanding dengan penapis yang disediakan daripada EFB yang tidak dirawat. Bahagian kedua melibatkan kajian tentang prestasi penuras. Kadar aliran, kedalaman penapis, kandungan kitosan dalam serat dan kepekatan influen dikaji dengan menggunakan Kaedah Permukaan Sambutan. Keputusan menunjukkan bahawa media penapis kitosan hanya mampu menapis 28.14 % untuk pepejal terampai total, 29.86 % untuk minyak dan gris, dan 8.13 % untuk keperluan oksigen kimia. Kandungan kitosan dalam gentian adalah faktor yang paling penting dalam rawatan POME. Penapis dengan kedalaman yang lebih rendah dapat mengeluarkannya zarahan dengan cepat tetapi disebabkan oleh ketipisan media, ia tidak dapat menyingkirkan sebahagian besar daripada zarah dalam tempoh masa yang panjang. POME dengan kepekatan influen yang lebih tinggi membolehkan kejatuhan tekanan yang lebih pesat semasa proses penapisan. Di samping itu, semua media penapis mengalami kehilangan kebolehtelapan dan mudah tersumbat. Ini menyebabkan penapis yang dikaji tidak boleh digunakan untuk proses penapisan yang mengambil jangka masa panjang.

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LIST OF ABBREVIATIONS

ANOVA	-	analysis of variance
BOD	-	biochemical oxygen demand
CCD	-	central composite design
COD	-	chemical oxygen demand
DOE	-	design of experiment
EFB	-	empty fruit bunches
NaOH	-	sodium hydroxide
NH^{3+}	-	positive charged amino acid
O&G	-	oil and grease
POME	-	palm oil mill effluent
RE	-	removal efficiency
RSM	-	Response Surface Methodology
S/N	-	signal-to-noise ratio
TSS	-	total suspended solids
WA	-	water absorption

LIST OF SYMBOLS

A	-	area of filter medium
α	-	specific cake resistance
β_M	-	medium resistance
β_0	-	constant coefficient
β_i	-	linear coefficient
β_{ii}	-	quadratic coefficient
β_{ij}	-	second order interaction coefficient
C_i	-	initial concentration of influents
g	-	gram
kg	-	kilogram
k_i	-	permeability of clean filter
k	-	permeability coefficient of the bed
L	-	thickness of filter media
ΔP	-	pressure drop across the medium
Q	-	volumetric flow rate
Re	-	Reynolds number
t	-	time of filtration
μ	-	viscosity of liquid
V_L	-	volume of liquid phase
W_b	-	mass of the sample after immersion
W_a	-	mass of the same sample before immersion

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CHAPTER 1

INTRODUCTION

1.1 Overview

Recently, due to a strong emphasis on environmental awareness worldwide, much attention has been brought to the development of recyclable and environmentally sustainable materials. Environmental legislation as well as consumer demand in many countries is increasing the pressure on manufacturers of materials and end-products to consider the environmental impact on their products at all stages of their life cycle, including recycling and ultimate disposal. Thus, a wide variety of renewable resources for the manufacture of new biodegradable products including structural/non-structural composites, moulded products, packaging, housing, automotive products and combinations of other materials, make it possible to explore new application areas such as geo-textiles, growths media, sorbents and filters media.

The use of lignocelluloses biomass as filter media is gaining more attention. Renewable resources such as lignocelluloses fibres have potential for utilisation in water and wastewater treatment applications. Their surface chemistry and large surface area should make lignocelluloses fibres ideal as filters. Filtration is a common process in wastewater treatment. Normally, the filtration efficiency usually depends on the materials used for filter. For physical types, the fibre-based filters can be used to remove suspended solid from water. The physical types of fibre filters can be in several forms. Fibres can be made into webs or mats, or packed into a column or chamber. Webs or mats increase the surface area of the filter and stabilize hydraulic pressure. The suspended solids are physical captured and held in the webs until the filters are cleaned. For chemical types, the fibre-based filters can also remove dissolved inorganic ions, organic chemicals, and other soluble contaminates from water. Several attempts have shown that fibrous media in the form of non-woven filters have been used extensively in water treatment as pre-filters or to support the medium that does the separation (Han *et al.*, 2003). Some studies also have been conducted on the wastewater treatment (Riahi *et al.,* 2009) or potable water treatment (Kim *et al.,* 2007) with depth filters, in which the packing material is an ensemble of fibres.

Some studies also show that the fibre can be modified to selectively remove desired contaminant (Kim *et al.,* 2007). Unmodified lignocelluloses fibres have great potential to absorb heavy metal ions. However, chemical modification techniques can potentially be used to enhance both heavy metal and oil sorption properties. Opportunities also exist for use lignocelluloses fibre filters in capturing dyes and nutrient from industrial processes. Several studies have tried to use low-cost, easily available materials for removal of metals (Lee and Rowell, 2004; Abia and Asuquo, 2007), dyes (Shukla and Pai, 2005) and nutrients (Karageorgiou *et al.*, 2007) from domestic and industrial wastewater under different operating conditions.

The impetus for the present study was a study of oil sorption by Rattanawong *et al.* (2007). They had modified oil palm empty fruit bunch fibres (EFB) by silylation agents to enhance the sorbability of oil emulsified in water. EFB composed of lignocelluloses material consisting of cellulose, hemicelluloses and lignin usually account for 65-70 percent of the plant's dry weight basis, which are major components of plant fibres (Han and Rowell, 1997). These compositions are hydrophilic, namely rich in hydroxyl groups, which are responsible for moisture sorption through hydrogen bonding. The hydrophilic properties must be modified before being utilised for oil removal. Fibre modification was conducted by leaching out surface contaminants resulting in an open porous surface. Results show that the sorption of oil which was emulsified in water on fresh and silylated EFB fibres fit Temkin and Langmuir isotherms, respectively.

One benefit of using lignocelluloses materials for filtration is their biodegradability after the filtration is disposed of. Most fibre material, especially agricultural fibres, will degrade in contact with moisture of water; some lignocelluloses material will degrade within days or weeks. Nature is programmed to recycle lignocelluloses in timely way through biological, thermal, aqueous, photochemical, chemical, and mechanical degradations. Completely biodegradable and environmentally-friendly green filter media can provide a more straightforward path for composting as the fibre and matrix does not require separating. In other industries, such as composite application, it is necessary to interfere with the natural degradation processes in order to produce composites with long services for as long as possible. However, in natural filter media application, degradation is not an issue.

1.2 Problem Statement

The utilisation of renewable resources in commercial filter media may potentially lead to the reduction of environmental pollution due to their biodegradability. Most of the currently available filter media in the market are made of synthetic polymers such as formaldehyde, polypropylene, polyethylene, rayon and nylon. Many of these polymers and fibres are petroleum-based and do not break down in nature after disposal. The end-of-life disposal of these filter media is becoming critical and expensive. Although there are some efforts to recycle and/or reuse the disposed filter media, most of them end up in landfills while some are incinerated, which is expensive and emits environment pollutants. However, at present the filters market still dominated by synthetic materials. Natural fibres are used to a much lesser extent, but their potential in such applications is promising.

However, the engineering challenge in replacing conventional synthetic filter media by biodegradable materials is to design materials using biodegradable raw materials that are fully functional and structurally stable during filtration process, yet are susceptible to environmental degradation upon proper disposal. The ultimate challenge is to do so in a cost-effective and environmentally-friendly manner in order to win public and political acceptance and still compete with the existing dominating materials in the market. The challenge is more pronounced where very limited research has been carried out on the utilisation of lignocelluloses biomass into filter media. One of the limitations of filter media from lignocelluloses biomass in wastewater applications is the lack of understanding of properties of filter media and filtration variables when using the media, and the relationship between them. The two properties must satisfy performance criteria before being considered for wastewater applications.

Another limitation of using lignocelluloses biomass as filter media in wastewater application is the lack of understanding in predicting the flow of liquids in filtration systems. According to English (1997), the Kozeny-Carman equation can be used to accurately predict the flow of liquids in simple systems; however, it has limitations when randomly oriented, highly variable, agro-based materials are considered. Natural fibre is different from synthetic fibre such as glass and nylon fibre. Researchers can calculate the specific surface area and specific volume of any synthetic fibre with the help of microscopic measurements whereas it is not possible in the case of natural fibres. Natural fibres are irregular in shape and are poorly defined. Furthermore, natural fibres have a lumen that does not contribute to the flow of water through it. Natural fibres swell in water due to their porous cell walls and if pressure is applied, the fibres collapse and form a ribbon-shaped structure.

Another prominent challenge is where very limited research has been carried out in the development of filter media without using synthetic adhesives or binders. The adhesives used in commercial products are usually synthetic polymer resins, based on the condensation reaction of formaldehyde with phenol, urea, resorcinol or melamine (Kupreev, 2012). With increasing concerns about the effects of emissive formaldehyde on human health, several efforts have been made to reduce or replace the phenol and formaldehyde content in adhesive formulations (Liu and Li, 2007). The interest in “green” adhesives from natural materials has also arisen due to the increasing concerns about the long-term supply of petrochemical resources. This has been coupled with an increasing drive for sustainability and environmental awareness that is related to the disposal of used filter media. It is therefore timely to review the current state-of-the-art in this area to promote sustainability that meets the needs of the present without compromising the ability of the future generations to meet their needs.

Chitin is widely distributed in nature and is one of the most abundant biopolymers. Today, chitosan is used widely in the wastewater industry (Sye *et al.*, 2008) and the health and medical sectors (Jayakumar *et al.*, 2010). Chitosan is used as a paper sizing or additive to improve paper's mechanical properties and water resistance (Li *et al.*, 2004). Moreover, it has been used for improving bonding properties of the particle board (Umemura *et al.*, 2009) and bio-composite (Julkapli *et al.*, 2008). Both studies elucidated the effects of the addition amount of chitosan on the physical and mechanical properties of the materials.

As reported by Umemura *et al.* (2005), chitosan and wood would create a certain affinity and intermolecular interactions due to chitosan's polycationic and wood's polyanionic nature. The study found that chitosan has good bonding properties with fibres, and reacts with reducing sugars such as glucose and xylose. However, no literature ever reported the application of chitosan as a binder in fabrication of filter media from lignocelluloses biomass.

EFB fibre are natural, inexpensive, and environmental friendly material available in enormous quantities thrown away as wastes after oil extraction processes, which occupies large areas in palm oil mill premises. Because of their low cost, these materials can be profitably used as alternatives or complements to the more commonly used methods for effluent treatment. The EFB fibre filter could be a potential technology for primary treatment of POME. The reason for developing filter media for wastewater application as the target is that the industry is actively seeking an alternative material from renewable resources. Due to their 3-dimensional network structure and large surface area, filter media from EFB fibres are feasible to be used as the filter to remove suspended solids, oil and grease and some organic compounds from POME.

This non-woven media is composed of randomly oriented fibrous fibres and will provide a one step separation as a substitute for conventional processes comprising chemical addition, flocculation, sedimentation, and sand filtration. As far as we know the incorporation of chitosan into EFB as filter media has not yet been reported. The aim of this study was to investigate the possibilities of developing a simple and inexpensive filter media from EFB as a porous medium for the primary industrial wastewater treatment. With no known prior investigation of using filter media for primary treatment, this is an empirical 'proof-of-concept' and feasibility study.

1.3 Objectives of Study

The main objective of the present research was to develop and test the performance of non-woven filter media using inexpensive empty fruit bunch fibres (EFB) and chitosan solution. The objectives of the research are as listed below:

i. To develop and fabricate non-woven filter media from EFB
ii. To characterize the newly developed non-woven filter media.
iii. To evaluate the permeability of the non-woven filter media using the Taguchi method.
iv. To evaluate the performance of non-woven filter media for pre-treatment of POME
v. To study the performance of the non-woven filter media for pre-treatment of POME using Response Surface Methodology.

1.4 Scope of Research

The scopes determine the extent of investigations in achieving all objectives mention in the previous section. Thus, to achieve the objectives, the following scopes have been undertaken:

i. Fabrication of binder-less empty fruit bunch filter media and chitosan-filled filter media were developed using treated and treated EFB fibres using wet lay-up method. Pre-treatment of EFB prior filter fabrication involved the extraction using sodium hydroxide, diethyl ether, ethanol and hot water.

ii. Non-woven filter media characterisation based on packing density, porosity, optical image, SEM, FTIR, thermal properties, tensile strength and water absorption properties.

iii. Permeability studies were carried out using filter media from untreated fibres. Taguchi method was used to design the permeability experiments. Permeability of the filter media using treated fibres were then tested using the optimum filter design from Taguchi design.

iv. The pre-treatment of POME using chitosan-filled filter media were carried out to evaluate the removal efficiency of total suspended solid and oil and grease removal efficiencies. A series of filtration process were carried out with different parameters of flow rate, chitosan loading, filter depth and influent concentration on filtration efficiency and pressure drop of filters. Permeability study was carried out using used filter media to study the behaviour of permeability reduction phenomena.

v. Response Surface Methodology was carried out using STATISTICA software to generate an experimental design for palm oil mill effluent pre-treatment, followed by a prediction model with subsequent investigations on the model's adequacy.

1.5 Significance of Research

Utilising the agricultural by-products in filter media is an ideal solution that may potentially lead to the reduction of environmental pollution caused by the palm oil mill industry. In Malaysia alone, the palm oil mill industry is producing huge quantities of oil palm biomass of about 90 x 10^6 tons of lignocelluloses biomass each year, of which 40 x 10^6 tonnes are in the form of EFB (EFB), oil palm trunks (OPT) and oil palm fronds (OPF) (MPOB, 2003). The EFBs represent about 9% of this total solid waste production (Alam *et al.*, 2008). Among the above fibre source in an oil palm tree, EFBs have the potential to yield up to 73% fibres and hence it is preferable in terms of availability and cost (Rozman *et al.,* 2000).

The palm oil industry has to dispose of about 1.1 tonnes of EFB per every tonne of oil produced. Some quantity of this highly cellulosic material is currently used as boiler fuel (Sreekala *et al*., 1997) in the preparation of fertilisers or as mulching material (Singh *et al.*, 2012) whereas a major portion is left in the mill premises itself. When left in field, these waste materials create a good habitat for insects and pests, thereby causing severe environmental problems (Sreekala *et al*., 1997, Law *et al.,* 2007). A sound practice will have to be engineered to manage the agro-wastes. Until today, a number of utilisation techniques have been developed to exploit these biomass residues in a rational way. These techniques include the production of particleboards, fibreboard (Ramli *et al.*, 2002), pulp (Chen *et al.*, 2004), paper (Ashori *et al.*, 2008), composites (Rozman *et al.*, 2004) and bio-composites (Avérous an Le Digabel, 2006). However, more than 60% of EFBs still remain unused. Thus, finding a beneficial utilization for the EFBs will surely alleviate environmental problems related to the disposal of oil palm waste.

EFB are currently utilised in a wide array of industries, but their applicability to environmental processes is still under only limited study. No research has been conducted to investigate the production of filter media from EFBs for the adsorption oil and grease from wastewater effluent. Because the filter media integrates the functions for both the deep filtration and mechanical screen, EFB filter media can potentially be used in the wastewater treatment industry. Thus, utilising the EFB in filter media is an alternative solution that may potentially add value to the by-products of the palm oil mill industry in wastewater application. The processing, characterisation, properties and applications of this promising class of high strength filter media materials is presented in detail in the thesis.

In addition, compared to synthetic fibres, EFB fibre delivers many advantages as inexpensive, reliable, durable, and can be easily further decomposed because of its biodegradability. Growing environmental awareness and increasing interest in sustainable material concepts have led to the development of bio and green filter media for wastewater applications. Thus, the present research was to prepare biodegradable filter media using EFBs and biodegradable adhesives. The newly developed filter media described in this research represent an approach to formulating green filter media that aim to reduce environmental pollution at the end of their useful lives. The adhesive or binder used for the web bonding was chitosan solution that was obtained by dissolving chitosan powder in aqueous acetic acid. With the chitosan treatment, filter media is a much better material to use in wastewater application, and its organic nature makes chitosan safe and environmentally friendly.

1.6 Outline of the Thesis

This thesis begins with an overview of the importance of sustainability development in filter media in Chapter 1. This chapter also highlights the objective and the scopes implemented in order to achieve it.

Chapter 2 begins with a review of lignocelluloses and their current applications; and several lignocelluloses surface treatment methods. In this chapter, previous work of researcher is also reviewed, which consists of researches on EFB and its current status of utilisation. This chapter also stated the filtration theories, which is very important in understanding the process itself. Previous works on chitosan application as a binder were also included in this chapter. The theories permeability of non-woven filter media was also discussed at the end of the chapter.

Chapter 3 describes procedures, equipment and chemicals that have been used starting from lignocelluloses fibre and binder preparation, filter media fabrication and filter media characterisation. Chapter 3 also reports the physical, thermal and mechanical properties of newly developed non-woven filter media. This chapter also involved permeability study using the design of experiment approach based on the Taguchi method in order to minimise the variation of the fabricated filter media. The last part in Chapter 3 involved a confirmatory final phase using the best combination of design parameters indicated by the analysis of the Taguchi design.

Chapter 4 begins with a review of Response Surface Methodology. The next part of this chapter describes the methods employed in a case study of palm oil mill effluent treatment, including wastewater sample preparation, analytical analysis and filtration experimental procedures. The effects of different parameters on the performance of newly developed non-woven filter media including of influent concentration, chitosan loading in the fibres, depth or thickness of filter and filtration flow rate were evaluated in this chapter. In the next part of Chapter 4, the process parameters affecting the filtration process were studied with a minimum number of experiments by using Design of Experiment (DOE) coupled with Response Surface Methodology (RSM). The design has identified and addressed the effect of important variables on the suspended solids, oil and grease and COD removal. The models generated in order to obtain the optimum conditions corresponding to each respond are properly explained and justified.

Chapter 5 concludes the research work and summarises all relevant findings generated from the study. This chapter also offers some recommendations for future work.

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