Faculty of Engineering

ANALYSIS AND KINETIC STUDY ON EXTRACTION PROCESS OF CELLULOSE FROM PLANT-BASED MATERIAL

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Bachelor of Engineering with Honours

(Chemical Engineering)

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APPROVAL SHEET

This report which entitled " **Analysis and Kinetic Study on Extraction Process of Cellulose from Plant-Based Material**" was prepared by Nurshuhada Binti Garip (59055) as a partial KNC 4344 Final Year Project course fulfillment for the Degree of Bachelor of Chemical Engineering is hereby read and approved by:

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ANALYSIS AND KINETIC STUDY ON EXTRACTION PROCESS OF CELLULOSE FROM PLANT-BASED MATERIAL

NURSHUHADA BINTI GARIP

Report is submitted to

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Specially dedicated to my amazing parents, for their endless support and unconditional love in every single day to motivate me to finish what I have started in this path of the journey I chose.

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ABSTRACT

This study aimed to evaluate the extraction of cellulose from plant-based materials including the analysis on parameters affecting the extraction process and the kinetic study on the yield of cellulose extracted from lignocellulosic biomass. Cellulose is the most abundantly available organic matter on earth, which can be derived from various sources. Lignocellulosic biomass materials are receiving increased attention as an economical, abundant, and renewable alternative for the production of various value-added products. Various conversion technologies are used for the production of value-added products from biomass, including chemical treatment, physical treatment, etc. Pretreatment plays a critical role in the yield of the products. The effect of extraction parameters on the pretreatment of plant fibres was described in this study. The results showed that temperature, alkali treatment, and extraction time have a significant effect on the yield of cellulose obtained. Also, the applicability of different mathematical models (Peleg and Page models) to describe the kinetics of the extraction process of cellulose from plant materials was studied as well. The study was performed in order to select the most suitable model to describe the kinetics of the extraction process of cellulose. Mathematical modelling is an extremely useful engineering tool that helps to study the effect of various parameters together with making behaviours predictions for future applications. The mathematical models applied showed a good agreement with the experimental result, and Peleg model showed the best fitting results compared to other models. Validation of the kinetic models was based on the determination of coefficient (R^2), and root mean square error (RMSE). Peleg's model with the highest R^2 (0.998) and the lowest average value of RMSE (0.2265) was determined to be the most suitable model to describe the extraction process of cellulose.

Keywords: Mathematical models, cellulose, extraction

ABSTRAK

Kajian ini bertujuan untuk meniali proses pengekstrakan selulosa dari bahan berasaskan tumbuhan dan mengkaji pengaruh parameter pengekstrakan terhadap hasil selulosa yang diekstrak dari biomas lignoselulosa. Selulosa adalah bahan organik yang paling banyak terdapat di muka bumi yang berasal dari pelbagai sumber. Bahan biomass lignoselulosa mendapat perhatian yang lebih tinggi sebagai alternative yang ekonomik, berlimpah, dan boleh diperbaharui untuk pembuatan bahan yang bernilai dari biomas. Berbagai teknologi penukaran digunakan untuk pembuatan produk bernilai dari biomas, termasuk rawatan kimia, rawatan fizikal, dan lain-lain. Pra-rawatan memainkan peranan penting terhadap hasil produk. Kesan parameter pengekstrakan pada serat tumbuhan dijelaskan dalam kajian ini. Hasil kajian menunjukkan bahawa suhu, kepekatan alkali, dan masa memberikan pengaruh yang signifikan terhadap hasal selulosa yang diperoleh. Juga, penerapan model matematik yang berbeza (model Peleg dan model Page) untuk menggambarkan kinetik proses pengekstrakan selulosa dari bahan tumbuhan juga dikaji. Kajian in dilakukan untuk memilih model yang paling sesuai untuk menerangkan kinetic proses pengekstrakan selulosa. Pemodelan matematik adalah alat kejuruteraan yang sangat berguna yang dapat membantu mengkaji kesan pelbagai parameter dan membuat ramalan tingkah laku untuk aplikasi masa hadapan. Model matematik yang digunakan menunjukkan persetujuan yang baik dengan hasil eksperimen; dan model Peleg hasil yang terbaik berbanding model lain. Pengesahan model kinetic dibuat berdasarkan penentuan pekali (R^2) dan ralat kuasa dua punca min (RMSE). Model Peleg dengan nilai R^2 tertinggi (0.998) dan nilai purata terendah RMSE (0.2265) ditentukan sebagai model yang paling sesuai untuk menggambarkan proses pengekstrakan selulosa

.

Kata kunci: Model matematik, selulosa, pengekstrakan

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ABBREVIATIONS

RMSE	Root mean square error
R^2	Correlation coefficient
NaOH	Sodium hydroxide
SEM	Scanning electron microscopy
FAO	Food and Agriculture Organization
OH	hydroxide
H_2O_2	Hydrogen peroxide
NaOCl	Sodium hypochlorite
NH3	Ammonia
$Ca(OH)_2$	Calcium hydroxide
H_2SO_4	Sulfuric acid
HCl	Hydrochloric acid
H_3PO_4	Phosphoric acid
SO_2	Sulfur dioxide
O_2	Oxygen
O_3	Ozone
CHF	Corn husk fibre
UNIMAS	Universiti Malaysia Sarawak

NOMENCLATURE

%	Percent
mm	Millimeter
µm	Micrometer
wt	weight
nm	nanometer
cm	centimeter
kg	kilogram
ha	hectare
m	meter
MJ	Megajoule
GPa	GigaPascal
MPa	MegaPascal
m^3	Cubic metre
g	gram
°C	Degree celcius
hr	hour

CHAPTER 1

INTRODUCTION

1.0 Introduction

This section will briefly explain the background of the study, problem statement, research questions, research hypothesis, research gap, research objectives, and the scope of study of this research project.

1.1 Background of the study

Today, the usage of the bio-based industrial residues has been increased continuously due to the increasing number of industrial bio-residues and the rising of their management. The need for products and processes in line with environmental criteria has inspired many types of research to use sustainable sources of raw material. The attempt to utilize the byproduct of the main food crop as a source of fibre will be substantial as the increasing world population will need more effective land use to provide food as well as clothing to those who are in the poorer part of the world (Reddy & Yang, 2005). Various lignocellulosic fibres like sisal, ramie, flax, pineapple, hemp, etc. have earned significant research importance for at least two decades due to their biodegradability, sustainability, high strength, and availability in vast quantities at a fair price (Hong et al., 2016). However, work on the use of agricultural waste such as rice, corn, wheat, banana, etc. to extract high-quality vegetable fibres has hardly been published (Yilmaz et al., 2014; Kambli et al., 2018). Waste from agriculture product often disposed of using open burning, which will pose health and environmental hazards. A scientific discovery of such agricultural waste, including waste derived from underused fibres would not only be useful in providing a livelihood for the rural community because of its added value. However, it will also lead to the arrest of local pollution due to the disposal of such large quantities of available agro-biomass. Yet another environmental advantage is that the products made from such agricultural byproducts can be made 100% bio-degradable.

Lignocellulosic biomass is the most plentiful renewable biomass on earth, with an estimated amount of about 200 billion tons of lignocellulosic biomass from wood, and agricultural waste worldwide (Yang et al., 2008; Das et al., 2016). Thus, it is essential for the development of a suitable scientific and economical method for recycling and reusing of these agricultural wastes to minimize the environmental problems associated with their build-up (Motaung & Linganiso, 2018). Naik et al. (2010) reported that currently, extensive work had been carried out globally to discover attractive chemical transformations to tum biomass into valuable products and to establish commercially viable methods for these changes. Natural cellulosic fibres come from different part of the plant. The fibres primarily categorized as seed fibres, leaf fibres, stem or bast fibres, etc. Such fibres may be from plants grown primarily for fibres or from plants in which the fibres are known primarily as a byproduct such as a coconut (the fibres sometimes referred to as coirs), pineapple, banana, sugarcane, etc. Lignocellulosic biomass consists of cellulose, lignin, hemicellulose, along with other minor elements. Between this cellulose is the most plentiful renewable organic matter produced in the earth and broadly distributed in plants, various animals, and to a lesser extent of algae, bacteria, etc. (Das et al., 2016).

Cellulose is a polysaccharide made of D-glucose linked through β-1,4-glycosidic bonds, and it is promising raw material for the production of valuable chemical substances, including cellulosic-ethanol hydrocarbon, and starting materials for polymer production. (Ragauskas et al., 2006). Cellulose is a common natural polymer, and it is a semicrystalline polymer having crystalline and amorphous regions in varying proportions dependent upon the species of the plant. Cellulose structures the backbone of the fibres in natural fibres, whereas lignin, as well as hemicellulose, primarily function as a glue to give structural support; they could be eliminated substantially by chemical treatment developed in the laboratory. Besides, cellulose is a biodegradable polymeric material that is linear and has a high molecular weight. Also, it can be found widely in a plant. The source can range from wood and even to agricultural waste. Due to this reason, the cell dimensions and the chemical compositions are depending on particular plant, their origin, and extraction methods (Eichhorn et al., 2010; Siqueira et al., 2010).

Until now, many researchers have reported on the extraction of cellulose from diverse natural source materials, especially in agricultural wastes, for instance, pineapple leaf (Santos et al., 2013), rice husk (Johar et al., 2012), wheat straw (Alemdar & Sain, 2008), mengkuang leaves (Sheltami et al., 2012), mango seed (Henrique et al., 2013), etc. The peculiar hierarchical architecture of natural cellulose comprising of crystallites and nanoscale fibrils makes it possible to extract nanoconstituents using chemical and mechanical methods, or by combining these two techniques. Scientist as well as researchers using different methods for extraction of cellulose from plant-based material, including alkali/acid treatment.

Solid-liquid extraction is a method that provides products that are commonly used in the food industries, cosmetics, pharmaceutical industries, as well as those used for environmental purposes. The latter has led to several scientific studies focused on the process kinetics (Bucic-Kojic et al., 2007), process design (Simeonov et al., 2017), as well as the yield of extraction (Bucic-Kojic et al., 2007). Abundant literature exists for the extraction of cellulose from plant-based material. However, there is a limited existing report available regarding the extraction kinetics of cellulose from plant-based materials. Therefore, there is a need to investigate the extraction kinetics and what drives it. Mathematical modelling is an extremely useful engineering tool which helps to explain a system and study the effect of various parameters together with making behaviours predictions for future application (Wright, 2015). Thus, this work will explore the suitability of different mathematical model used to evaluate the extraction process of cellulose from plant-based materials.

1.2 Problem Statement

The use of lignocellulosic natural fibres has become an attractive reinforcement in economic and ecological terms for polymer composites over the past few years. Yang et al. (2015) mentioned that the risen in the environmental awareness of the environment had given rise to interest in the research and development of both biodegradable and high-performance materials. Furthermore, increased awareness of the depletion of non-renewable resources and our eventual reliance on renewable resources has arisen (Shalwan & Yousif, 2013). Natural fibres are now emerging as viable alternatives to synthetic fibres either alone or as composite materials for diverse applications, including automotive components, biomedical applications, building materials, as well as packaging industries. Plant fibres are the major natural sources of cellulose. Cellulose is the organic matter that is most commonly found on earth. Globally, agricultural produces millions of tons of agricultural byproduct yearly including the wastes, which some of them are potential contributors of environmental pollution. Unused agricultural produces may further be processed such as for cellulose extraction. Cellulose is a fascinating and almost inexhaustible sustainable natural polymer that has been used for thousands of years in the form of fibres or their derivatives for a wide range of materials and products

There are some works on cellulose extraction found in the literature, but in depth evaluation on the cellulose extraction from plant-based materials including analysis of parameters affecting the extraction as well as the modeling for the extraction is considered limited. Hence, there is a necessity to study in depth on these areas. The mathematical modelling is an effective tool for equipment simulation, design, optimization, as well as control, allowing a theoretical description of the process. In scientific literature, the processes of solid-liquid extraction from a variety of natural materials are simulated and described via various mathematical models. Knowledge and understanding of the kinetics of natural product extraction are great economic importance as the extraction process is an important industrial operation. Empirical kinetic models mathematically explain variations in the amount of bioactive compounds in either plant material or liquid extract over time. These kinetic models are usually more straightforward compared to the physical ones but then are still appropriate for engineering purposes (Rostami & Gharibzahedi, 2017). Therefore, this research aims to select the appropriate kinetic model for the extraction of cellulose from plant-based materials based upon its accuracy of fitting the experimental data obtained and examined the effect of extraction parameters on the extraction kinetics.

1.3 Research Questions

The research questions to be addressed in this study are:

a) What is the yield of cellulose from different types of plant-based materials?
b) What is the effect of varying the parameter of the extraction process to the yield of cellulose?
c) What is the best mathematical model that may be used to describe the kinetics of the extraction process?

1.4 Research Hypothesis

The research hypothesis to be addressed in this study are as follows:

a) Different types of plant-based material yield a different amount of cellulose.
b) The yield of extracted cellulose increases with the temperature, alkali concentration, and extraction time.
c) The best mathematical model fits well the extraction process of cellulose with highest value of correlation coefficient (R^2), and lowest value of root means square error (RMSE).

1.5 Research Gap

Table 1.1 shows some recent studies on the extraction processes of different types of materials. Previous research has shown that various mathematical model has been used to predict the output of extraction process from different types of materials; further research is necessary to use a mathematical model to predict and evaluate the extraction process of cellulose from plant materials, the gap of research that will be studied in depth in this work.

Table 1.1 Research Gap

Research Scope	**Research by**
a. Studied of mathematical modelling for extraction of flavonoids from *Clinacanthus nutans* leaves (Belalai Gajah). In this research, the maximum yield and time of exhaustive flavonoids extraction were determined by using Peleg's model.	(Mohd Fazil et al., 2016)
b. Studied the applicability of various mathematical models (Page, Logarithmic, and Peleg models) to explain the kinetics of solid-liquid extraction process from soybeans. In this study, parameters like temperature, extraction time and type of solvent were tested.	(Jokic et al., 2010)
c. Study the influence of temperature as well as a solid-liquid ratio on the total polyphenols yield from grape seeds. The extraction kinetics were estimated using modified Peleg's model	(Bucic-Kojic et al., 2007)
d. Research on the extraction of pectin from dragon fruit peels by ultrasound-assisted extraction method. Five mathematical models have been applied to investigate the kinetics of the extraction process. Temperature and time were found to have a major effect on the yield of pectin.	(Lin et al., 2018)
e. Kinetic study on the extraction process of cellulose from plant-based materials.	Research gap

1.6 Research Objectives

There are several objectives to be achieved in this research. The objectives of this research study are listed below:

a) To study and compare the yield of extracted cellulose from different types of plant-based materials.
b) To investigate the effect of temperature, NaOH concentration, and extraction time on the yield of extracted cellulose.
c) To evaluate and choose the best mathematical model used for the extraction process of cellulose.

1.7 Scope of the Study

The scope of the study mainly focused on comparing the yield of cellulose from different categories of plant fibres. Cellulose is an abundant renewable material and extracted from various plants materials. The growing interest in lignocellulosic fibres is mainly due to the series of advantages such as biodegradability, low cost, as well as low density. In this work, the effect of extraction parameters on extracted yield of cellulose such as temperature, alkali concentration, and extraction time will be analyzed. Besides, the extraction kinetics of cellulose will be evaluated by comparing two mathematical models, Page and Peleg models that are commonly used in the extraction process.

1.8 Summary

In summary, this chapter discusses the background of the research study, problem statement, research objective and scope of the study. The research gap has also been included to show the continuity of research in similar area.

CHAPTER 2

LITERATURE REVIEW

2.1 Natural plant fibres

Nowadays, natural plant fibres have become a viable option for the most utilized fibre in composite technology (Djafari Petroudy, 2017). Natural plant fibre has excellent possibility as a replacement for the non-renewable energy sources material, such as petroleum in numerous applications. Besides, natural plant fibre also has established itself as the highly acceptable option reinforcement for fibre composite products. Asasutjarit et al. (2007) stated that the key drivers for their implementation as alternatives for conventional reinforcements in composite materials are the combination of their attractive mechanical as well as physical properties, and also their environmental benefits. Moreover, natural fibres are the ultimate green products and need less energy to generate in comparison to the most common synthetic reinforcing fibres (Mobasher, 2012).

The main characteristic that distinguishing the plant cells from cells in animals is the rigid cell wall that surrounded the cells of the plants. For some cell types, the cells walls are expanded in order to have a superior mechanical property of the cell walls, providing the required structural strength of the plants. Djafari Petroudy (2017) stated in his study that different plants have different dimensions of the fibres. Still, the general shape is most frequently extended with the diameter within a range of 15-30 µm and length in the range of 1-35 mm. Besides, fibres are hair-like material that are in discrete elongated pieces or are continuous filaments, which is like thread fragments, and they can be dipped into ropes, thread, or filaments. According to Chandramohan and Marimuthu (2011), natural fibre may be used as a part of material composites. **Figure 2.1** illustrates the classification of natural plant fibres. From the figure, there are six types of natural plant fibres, viz. leaf fibres