PROCESS SELECTION AND OPTIMIZATION OF CELLULOSE EXTRACTION FROM PALM KERNEL USING RESPONSE SURFACE METHODOLOGY

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**SCHOOL OF ENGINEERING
AND INFORMATION TECHNOLOGY
UNIVERSITI MALAYSIA SABAH**

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ABSTRACT

PROCESS SELECTION AND OPTIMIZATION OF CELLULOSE EXTRACTION FROM PALM KERNEL USING RSEPONSE SURFACE METHODOLOGY

Palm kernel cake (PKC) as one of the abundant high cellulose content oil palm by-products. Hence, PKC is significantly considered as potential resources for cellulose production. Numerous treatments have been developed in efforts to extract cellulose from wood and non-wood sources. However, efficient separation of cellulose from non-wood sources still constitutes as one of the major obstacles to extensive utilization of cellulose in industry. The present research was undertaken to select the best cellulose extraction method for PKC and followed by optimization of the selected method. The extractions of PKC cellulose were compared with the established pretreatments (hydrothermal treatment and alkali treatment) and three different delignification techniques (Organolsolv, Liquid phase oxidation (LPO) and Acidic sodium chlorite). Both of the treated residues were verified by FTIR as cellulose, yet with some contamination. Hence, process selection was conducted to select the best cellulose extraction method. LPO with 77 minutes of alkali treatment was selected as the best cellulose extraction process. Next, it was followed by the optimization of the LPO. The optimized conditions are ratio of H_2O_2 to PKM at 10.30 (g/g), 149min of reaction time, reaction temperature at 65°C and 12.5mg of $FeSO_4$ as catalyst for 24% of hemicellulose removed, 16% of yield cellulose based on the dry basis of PKC and 90.31% cellulose purity. Pre-identification size of PKC cellulose using GPC was carried out. It was discovered that PKC cellulose is α-cellulose with an intermediate molecular weight. For the analysis of organic acids content, formic acid and acetic acid formed as the dominant acids in the solution. LPO with alkali treatment is not only a green and efficient cellulose extraction method, it also produce valuable by-products. Hence, extraction of cellulose from palm kernel cake using LPO with alkali treatment is a great potential for new industrial process.

ABSTRAK

Kandungan sellulosa dalam hasil-hasil sampingan pertanian telah menggalakkan aktiviti penyelidikan menggunakannya sebagai alternatif sumber-sumber tenaga tambahan atau bahan kimia. Sisa isirung kelap sawit (PKC) merupakan salah satu hasil sampingan yang banyak dengan kandungan selulosa tinggi dari industri kalapa sawit. Ini dapat mempertimbangkannya sebagai sumber yang berpotensi untuk menghasilkan sellulosa. Terdapat banyak perawatan yang telah dibangunkan untuk mengekstrak sellulosa. Walaubagaimanapun, pemisahan sellulosa dari sumber-sumber tumbuhan dengan berkesannya masih merupakan halangan utama bagi mengunakan sellulosa dengan sepenuhnya dalam industri. Oleh itu, kajian ini telah dilakukan untuk mendapat satu kaedah pengekstrakan selulosa yang berkesan bagi Sisa isirung kelap sawit. Pencabutan bagi sisa isirong kelapa sawit selulosa yang telah dipertandingkan dengan gabungan prarawatan (rawatan air panas dan perlakuan alkali) dan tiga kaedah-kaedah *delignification* berbeza (Organolsolv, fasa cecair pengoksidaan dan natrium Acidic klorit). Sellulosa daripada sisa isirung kelap sawit telah disahkan oleh FTIR. Ini telah diikuti dengan proses optimal pengekstrakan sellulosa yang terpilih, fasa cecair pengoksidaan dengan alkali prarawatan dengan menggunakan RSM. Antara ini, fasa cecair pengoksidaan dengan alkali prarawatan pada 77 mins telah ditentukan sebagai gabungan pengekstrakan sellulosa yang terbaik. Optimasi telah dijalankan deangab kaedah yang terpilih. Proses parameter yang digunakan bagi mengoptimalkan fasa cecair pengoksidaan dengan alkali prarawatan ialah jumlah $FeSO_4$ digunakan, nisbah sisa isirung kelapa sawit kepada H_2O_2, suhu dan masa tindakbalas pengoksidaan fasa cecair. 24% hemiselulosa telah berjaya dibuangkan, 16% selulosa hasil dari PKC telah berjaya diekstrak dan 90.31% ketulenan selulosa berbandingan Organosolv dan berasid natrium klorit. Ciri- ciri sellulosa ditentukan dengan analisis Spektrum IR sellulosa sisa isirung kelapa sawit menggunakan FTIR dan pra-pengenalpastian saiz dengan menggunakan kromatografi cecair prestasi tinggi (HPLC). Analisis kandungan asid-asid organik dalam larutan rawatan pengekstrakan juga dilakukankan dengan menggunakan HPLC. Formik dan asid-asid Acetic telah didapati sebagai dominant asid-asid organik dalam larutan tersebut.

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LIST OF ABBREVIATION

ASAM	Alkaline Sulfite Anthraquinone Methanol
AGU	Anhydroglucose Unit
ANOVA	Analysis Of Variance
DMAC	N,N-Dimethylacetamide
DV	Viscometric Detector
RID	Refractive Index Detector
FTIR	Fourier Transform Infrared Spectroscopy
GPC	Gel Permeation Chromatography
HPLC	High Performance Liquid Chromatography
JIS	Japanese Industrial Standard
LiCl	Lithium Chloride
LS	Light Scattering Detectors
MALS	Laser Light Scattering And Macromolecular
min	Minute
mm	Millimeter
NMR	Nuclear Magnetic Resonance
PKC	Palm Kernel Cake
PKM	Palm Kernel Meal
THF	Tetrahydrofuran
UV	Ultraviolet

LIST OF SYMBOL

µ	Micro
C	Concentration
T	Temperature
t	Time
C_n	The n th carbon
w/w	Weight by weight
α	Alpha
m	Milli
v/v	Volume by volume
E	Exponentials

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Palm kernel cake (PKC) is one of the major by-products of palm oil processing industry, which is produced in sizable quantity annually. About 5670 billion tons of PKC is produced by the palm oil industry in the world in 2007 (USDA, 2008). The global production of PKC is continuously increasing due to the tremendous growth of the palm oil industry within the Southeast Asia region and Africa (PORLA, 2000) and the demand world wide.

Currently, most of the PKC is exported from Asia countries at low price to European Union (EU) countries and utilized as ruminant feed supplements for livestock such as cattle, dairy cows, goat, sheep and horses (Ng, 2004; Hishamuddin, 2002; Wan and Alimon, 2003; Siew, 1989). However, the utilization and absorption of PKC based feed by monogastric animals such as poultry, pigs and aquacultures is inhibited. Beta mannan content in PKC as anti-nutritive compound is the main obstacle in the poultry feed (Sundu and Dingle 2003; S. Abd-Aziz *et al.*, 2009) due to low digestibility and low metabolisable energy value. Therefore, improvement in quality of PKC is carried out to increase the digestibility and protein contain of PKC for poultry feeding (Iluyemi *et al.*, 2006; Ng, 2004; Kolade *et al.*, 2005; M Boateng *et al.*, 2008). Besides, some of PKC is disposed off by burning as fuel sources in industry (Kolade *et al.*, 2005). However, with increasing demand for renewable energy, PKC which has high calorific value and characteristics are also being processed into alternative liquid or gaseous form of renewable energy such as bioethanol, bioils and producer gas (Chew and Bhatia, 2008; Gutiérrez *et al.*, 2009).

PKC has a good potential as a sustainable resource for various industries. It contains significantly high percentage of lignocelluloses (Chew and Bhatia, 2008) with roughly 30% of cellulose content (Iluyemi *et al.*, 2006). Due to its basic high percentage cellulosic components, PKC is a potentially viable source of cellulose. The extracted cellulose can be further processed for ethanol production (Chen and Liu, 2007) and cellulose derivatives (Puspamalar *et al.*, 2006; Hasan and Nurhan, 2003), which is widely used as thickener, stabilizer agent and emulsifier. Besides, PKC can be converted to organic acids due to its high lignocellulose content (Lopretti *et al.*, 2000; Sundqvist *et al.*, 2006). These organic acids are produced as valuable by product during the cellulose extraction process. Various organic acids are produced such as formic acid, propionic acid, lactic acid and acetic acid during hydrothermal reaction(He *et al.*, 2008; Sundqvist *et al.*, 2006) and oxidation (Mae *et al.*, 2000), which commonly occur in cellulose extraction process to loosen and cleave the linkage of hemicellulose and lignin before cellulose becomes accessible. Currently, the organic acids are applied extensively as additives, acidulants, food preservatives, buffering agent, and potential as alternative antibiotic in poultry industry and bioplastic production. Hence, the extraction of cellulose and organic acids as by-products will definitely enhance the current usage of PKC.

In addition, PKC is also rich in carbohydrate (Kolade, *et al.*, 2005), has relatively high protein content and essential amino acid profile (Iluyemi *et al.*, 2006). Analytical data shows that PKC contain about 20% of protein (Ng, 2004; Iluyemi *et al.*, 2006; M. Boteng *et al.*, 2008). This palm kernel protein can be extracted and purified to be used as animal feed supplement. However, with the current trend of green chemistry, palm kernel protein is utilized for production of plant protein based resin. The resin is non-toxic and formaldehyde-free. Soy protein is used as one of the alternatives to conventional resin (Huang and Netravali, 2007; Lodha and Netravali, 2002 & 2005). The polar amino acid content can improve the mechanical and thermal properties and moisture resistance of the wood glue (Huang and Netravali, 2007). However, the adhesive is not practical for commercial application due the high cost of soy protein isolate (Huang and Li, 2008). The high amino acid (84.5%) of protein from palm kernel meal (Perez *et al.*, 2000) compared to soy bean meal (97.3%) suggests that PKC protein is useful for production of formaldehyde free wood glue.

The study of utilization enhancement of palm kernel cake is a broad process; therefore the research here is focused on the extraction of cellulose from PKC. There are various benefits throughout the cellulose extraction from PKC. The extraction process not only produce cellulose but also organic acids as by-products as explained above. Before the cellulose extraction process, the research involved oil removal from PKC to enhance the efficiency of the extraction process. Besides, the extracted cellulose is edible and suitable for pharmaceutical purposes due to non-toxic chemical media, and is environmental friendly.

There has not been any research study on cellulose extraction from PKC before. Therefore, the research here involves comparative study with the performance of various hemicellulose removal and delignification techniques for the PKC cellulose extraction, and followed by optimization of selected combination with cellulose extraction process. The objectives of the research are shown below.

1.2 Research Objectives

The present research was undertaken to find the effective cellulose extraction method for PKC cellulose production.

The objectives of this research are:-

i. Comparison of the purity and yield of cellulose extraction from palm kernel fiber with the two established pretreatment (hot water treatment and alkali treatment) and three different delignification methods namely,
   a. Organolsolv
   b. Liquid phase oxidation
   c. Acidic sodium chlorite

ii. Optimization of process parameters for selected combination of cellulose extraction process.

1.3 Scope of Research

The scopes of the research are:-

i. To analyse the holocellulose, cellulose, lignin, ash content and protein content of oil free palm kernel

ii. To remove remaining oil from palm kernel using soxhlet extraction method with *iso-propanol* as solvent.

iii. Cellulose extraction from oil free palm kernel with combination of one of two established pretreatment (hot water treatment and alkali treatment) methods and one of the three different delignification methods (Organolsolv, Liquid phase oxidation and Acidic sodium chlorite).

iv. Verification of the extracted residues under the combine cellulose extraction processes methods using FTIR.

v. Comparison study of the combine cellulose extraction treatment techniques and pretreatment time using Response Surface Methodology.

vi. To study the process parameters: amount of $FeSO_4$, ratio of oil free palm kernel to H_2O_2, liquid oxidation reaction temperature and time for selected cellulose extraction process, liquid phase oxidation with alkali pretreatment.

vii. To analyze and study the structure cellulose components using Fourier transform infrared (FT-IR) and GPC.

viii. To determine the organic acids content of hot water pretreatment, liquid phase oxidation process treated solution using HPLC.

In order to accomplish this task, the following section outlines how the thesis work was done. The thesis is divided into five chapters (introduction, literature review, methodology, results and discussion, conclusions and future work).

1.4 Thesis Organization

Chapter 1 introduces the trend and awareness of utilization of agricultural residues as alternatives resources for supplementary energy sources or chemicals productions. The potential of PKC to be consider as bio-resource of raw materials due to its high cellulose content and abundance in Malaysia. The chapter also covers on the major problems which hamper the efficiency of cellulose extraction process. The research objectives and scopes are also laid down accordingly.

Chapter 2 presents the literature review on the chemistry and characterization of plant cellulose, plant cellulose sources and alternative resources, and applications of cellulose in industry. Further, the review on conventional cellulose extraction process and recent green cellulose extraction process methods has been discussed. An overview of the analysis for characterization of cellulose properties and statistical analysis with response surface methodology has also been discussed.

Chapter 3 discusses the research approach and methods used in this work. The research methodology has been explained in detail. The analytical methods used for determination of hemicellulose removed, yield and purity of cellulose have been elaborated. The instrumental analysis using of FTIR, HPLC and GPC has been explained in detail. Process selection of cellulose extraction for PKC was performed and followed by optimization of selected extraction process. Statistical analysis of the process selection and optimization is elaborated using Response Surface Methodology (RSM).

Chapter 4 is written about the results and discussions. The results and discussions were reported according to the sequence of the approach and methodology in chapter 3. The results and discussions start with the oil removal and composition analysis of oil-free PKC. Verification of the extracted residues by different combinations of cellulose extraction processes using FTIR is studied and compared and followed by the process selection for cellulose extraction. After the detailed analysis of the process selection, optimization of the selected cellulose extraction process with different process parameters was explained in detail. Characterization analysis using FTIR and GPC was then further discussed and followed by the analysis of organic acids content in process solution using HPLC.

Finally, Chapter 5 gives the conclusions based on the research findings from experimental tests performed for selection of the efficient cellulose extraction process and optimization of the selected extraction process with different process parameters. The chapter is also concluded with suggestions on possible future work.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In this research, extraction of cellulose from PKC is carried out by comparative study of various hemicellulose removal and delignification techniques and followed by optimization of selected cellulose extraction processes combination. Then the analytical analyses were conducted to identify and characterize the cellulose. Hence, the literature review aims to review the chemistry and characterization of plant cellulose, plant cellulose sources and alternatives resources, and applications of cellulose in industry with sufficient references to understand the importance of the research of cellulose extraction. Besides, the conventional and available cellulose extraction processes selected for this research have also been reviewed.

2.2 Chemistry of Plant Cellulose

The study of characterization and properties of cellulose are important to fulfill the various demands of functionality of cellulose production and to increase their industrial uses, since they have often been used and modified as raw materials in numerous industrial applications.

Cellulose is recognized as the major component of plant biomass. Generally, cellulose is a colorless, odorless and toxic-free biopolymer abundant in nature. The physical, chemical and biological properties of cellulose depend on its shape properties, such as its intrinsic form and its ease of deformability. Therefore, extensive effort has been devoted to chemical and structure studies on cellulose (Sun *et al.*, 2005).