OPTIMIZATION OF PRETREATMENT AND ENZYMATIC HYDROLYSIS OF PINEAPPLE LEAVES FIBER FOR REDUCING SUGAR PRODUCTION

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This thesis is dedicated to my beloved mother, *Khafizah Khalid,* who taught me that education is the most powerful weapon to change the world.

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ABSTRACT

Pineapple leaves fiber (PALF) are classified as an abundant lignocellulosic biofiber material in Asia including Malaysia. Conversion of PALF into reducing sugar has contributed to an alternative use of lignocellulose source especially in the production of value-added product such as amino acid, biofuels and vitamins. In bioconversion process, pretreatment of lignocellulosic biomass is important to enhance the accessibility of enzyme hydrolysis, which can increase the yield of reducing sugar. PALF have a very complex arrangement of structure thus proper study in pretreatment and enzymatic hydrolysis process is needed to obtain optimum yield of reducing sugar. In this study, PALF pretreated with sodium hydroxide (NaOH) contributed to the optimum yield of reducing sugar (11.65 mg/mL) which were higher compared to dilute sulphuric acid (8.11 mg/mL) and autohydrolysis pretreatment (6.50 mg/mL). The best pretreatment method of PALF using NaOH had been optimized using response surface methodology with Box-Behnken design. The optimum operating conditions of NaOH pretreatment were found to be at 2.43 % (w/v) of NaOH concentration, 87.22 °C temperature and 57.15 min pretreatment time which produced optimum yield of reducing sugar, 17.26 mg/mL. The optimized pretreated PALF was further hydrolyzed through enzymatic hydrolysis process using cellulase enzyme. The optimum operating conditions for enzymatic hydrolysis were found to be at 228.48 μg/mL of enzyme loading, 45 °C temperature and 69.06 min of hydrolysis time and produced optimum yield of reducing sugar, 108.74 mg/mL, with 84.12 % improvement before an optimization of enzymatic hydrolysis process. The kinetic studies of enzymatic hydrolysis process on pretreated PALF showed good correlation between the influence of enzyme concentration and substrate on the production of reducing sugar. Chrastil approach shows a good correlation (R^2) with all enzyme concentration at 50 μg/mL (0.97), 150 μg/mL (0.94), 200 μg/mL (0.94), 228.48 μg/mL (0.95), 250 μg/mL (0.92) and 300 μg/mL (0.90) while modified Michaelis-Menten model obtain $R^2 = 0.99$. In addition, it also shows that NaOH pretreatment on PALF was able to enhance the movement of molecules in pretreated PALF, thus enhance the reaction of enzyme into substrate. This study has proven that by optimizing the operating conditions of the pretreatment and enzymatic hydrolysis processes, the yield of total reducing sugar extracted from PALF could be enhanced.

ABSTRAK

Gentian daun nenas (PALF) diklasifikasikan sebagai bahan lignoselulosa yang banyak di Asia termasuk Malaysia. Penukaran PALF kepada gula penurun telah menyumbang kepada pengunaan alternatif sumber lignoselulosa terutamanya dalam penghasilan nilai tambah produk seperti asid amino, bahan bakar bio dan vitamin. Dalam proses penukaran-bio, pra-rawatan jisim bio lignoselulosa adalah penting untuk meningkatkan capaian penguraian enzim, dimana boleh meningkatkan penghasilan gula penurun. PALF mempunyai susunan struktur yang sangat kompleks dengan itu kajian yang baik terhadap proses pra-rawatan dan penguraian enzim diperlukan bagi mendapatkan hasil gula penurun yang optimum. Dalam kajian ini, PALF yang di pra-rawat dengan natrium hidroksida (NaOH) menyumbang kepada hasil optimum gula penurun yang lebih tinggi (11.65 mg/mL) berbanding asid sulfurik cair (8.11 mg/mL) dan pra-rawatan autohidrolisis (6.50 mg/mL). Kaedah pra-rawatan PALF terbaik menggunakan NaOH telah dioptimumkan dengan menggunakan kaedah gerak balas permukaan dengan reka bentuk Box-Behnken. Keadaan operasi yang optimum bagi pra-rawatan NaOH didapati pada kepekatan NaOH 2.43 % (w/v), suhu 87.22 ºC dan masa pra-rawatan 57.15 min yang menghasilkan gula penurun yang optimum, 17.26 mg/mL. Pra-rawat PALF yang telah dioptimumkan kemudiannya diuraikan lagi melalui proses penguraian enzim menggunakan enzim selulase. Keadaan operasi optimum untuk penguraian enzim adalah pada kandungan jumlah enzim 228.48 μg/mL, suhu 45 ºC dan masa penguraian 69.06 min dan menghasilkan gula penurunan yang optimum 108.74 mg/mL, dengan peningkatan sebanyak 84.12 % sebelum pengoptimuman proses penguraian enzim. Kajian kinetik proses penguraian enzim ke atas PALF pra-rawat menunjukkan hubungan yang baik antara pengaruh kepekatan enzim dan substrat ke atas pengeluaran gula penurun. Pendekatan Chrastil menunjukkan hubungan yang baik (R^2) dengan semua kepekatan enzim pada 50 μg/mL (0.97), 150 μg/mL (0.94), 200 μg/mL (0.94), 228.48 μg/mL (0.95), 250 μg/mL (0.92) dan 300 μg/mL (0.90) manakala model Michaelis-Menten yang diubahsuai memperoleh R^2 = 0.99. Tambahan pula, ia juga menunjukkan bahawa pra-rawatan NaOH ke atas PALF mampu meningkatkan pergerakan molekul dalam PALF pra-rawat, oleh itu meningkatkan tindakbalas enzim terhadap substrat. Kajian ini membuktikan bahawa dengan mengoptimumkan keadaan operasi pra-rawatan dan proses penguraian enzim, jumlah penghasilan gula penurun yang diekstrak dari PALF dapat ditingkatkan.

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LIST OF ABBREVIATIONS

ANOVA	-	Analysis of Variance
APEX	-	Ammonia Fiber Expansion
BBD	-	Box-Behnken Design
CCD	-	Central Composite Design
DNS	-	Dinitrosalicyclic Acid
FPU	-	Filter Paper Unit
H_2SO_4	-	Sulphuric Acid
HPLC	-	High Performance Liquid Chromatography
MM	-	Michaelis-Menten
NaOH	-	Sodium Hydroxide
OPF	-	Oil Palm Frond
OS	-	Organosolv
PALF	-	Pineapple Leaves Fiber
RSM	-	Response Surface Methodology
SEM	-	Scanning Electron Microscopy
TRS	-	Total Reducing Sugar

LIST OF SYMBOLS

^{o}C	-	Degree celsius
%	-	Percentage
h	-	Hour
µg/mL	-	Microgram per mililiter
mg/mL	-	Milligram per mililiter
w/v	-	Weight per volume
v/v	-	Volume per volume
v_o	-	Hydrolysis rate
E_o	-	Initial Enzyme Concentration
n	-	Structural Diffusion Resistance Constant
k	-	Rate Constant
V_{emax}	-	Maximal velocity
K_e	-	Corresponding Half-Saturation Constant

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Nowadays, lignocellulosic biomass has been known as a potential renewable and inexpensive energy source because of the abundant material in most countries especially in Asia (Chapla *et al.,* 2011). Large amount of lignocellulose, generated through forestry, agricultural and agro indutsries, are catogerized as waste that causing environmental pollution problem because of the large accumulation in nature. Therefore, the utilization of these lignocelluloses not only beneficial to produce value-added products, but also solved the issue of environmental problems. Lignocellulosic biomass can be utilized for the production of various value-added product suuch as biofuel, food aditives, animal feed, vitamins amico acids that produce from the fermentation process by using glucose as a sole substrate (Howard *et al.,* 2003).

Utilization of glucose as a main fermentable sugar in these fermentation to produce high-value product may not be economically viable in industry (Jia *et al.,* 2017). Therefore, lignocellulosic biomass with high in cellulose and hemicellulose content could save the cost by replacing the glucose by producing high yield of reducing sugar. Lignocellulosic biomass mainly composed of three major polymers which are cellulose, hemicellulose and lignin. The major source of lignocellulosic biowaste are agricultural waste such as rice straw, wheat straw, wood (hardwood), by-products left over from the corn milling process; corn fiber, annual and perennial crops, waste paper, and sweet sorghum (Visioli *et al.,* 2014). However, the composition of these constituent is different from one plant species to another and found that biofiber, which is the by-product produced from cultivation, shows a greater amount of cellulose. The composition, properties and structure of biofiber make them suitable for many usages included composite, textile, pulp and paper manufacture, fuel industry, chemicals and enzyme food (Reddy and Yang, 2005).

Pineapple leaves fiber (PALF) are one of the abundant biofiber products from pineapple cultivation., which is widely grown in Malaysia as well as Asia. Malaysia produces about 315.977 metric tons of pineapple for year 2013 which ranked it nineteenth pineapple producing country in the world (FAO, 2013; Agrofood Statistics, 2013). In addition, Yahya and Yusof (2013) reported that PALF consists about 70-82 % (w/w) cellulose content which is higher compared to other raw material for example barley straw, corn cobs, and wheat straw which were 42 % (w/w) (Kumar and Gayen, 2011), 33.7 % (w/w) (Nigam *et al.,* 2009) and 30.2 % (w/w) (Ballestros *et al.,* 2006) of cellulose content, respectively. Therefore, PALF have high potential to produce high yield of reducing sugar and become a new source, thus replace the non-renewable and expensive raw material in many industries (Asim *et al.,* 2015). However, to achieve high yield of reducing sugar, an ideal pretreatement process is required to enhance the accessibility of enzyme to converted polymer into simple sugar via enzymatic hydrolysis process.

In bioconversion, pretreatment of biofiber was a necessary step to disrupt the hemicellulose-lignin structure to enhance the cellulose accessibility to produce high yield of reducing sugar (Hong Ng *et al.,* 2013). An ideal pretreatment process should efficiently improve the enzymatic hydrolysis, consume lower amounts of chemicals, and produce fewer by-products/inhibitors (Karimi *et al.,* 2015). In pretreatment, there are few methods that have been done by many researchers including chemical, physical, physio-chemical (hot water), and biological pretreatment (Mood *et al.,* 2013). Among all the pretreatment method, chemical pretreatment (acid and alkaline) was the most common pretreatment applied for lignocellulosic biomass because of the ability to remove lignin and solubilize hemicellulose efficiently (Bradeur *et al.,* 2011). However, an ideal pretreatment was only required to alter the biomass structure as well as its overall chemical composition, hence enzymatic hydrolysis was needed to hydrolyse carbohydrate into simple sugar.

This research was conducted to produce high yield of reducing sugar from pretreated PALF via enzymatic hydrolysis. In this study, the different pretreatment methods were compared and investigated in order to alter the structural of lignocellulose to enhance the accessibility of enzyme to hydrolyse carbohydrate into simple sugar. Throughout this study, operating condition parameter for both ideal pretreatment and enzymatic hydrolysis were investigated to ensure the optimum reducing sugar was obtained.

Optimization of ideal pretreatment and enzymatic hydrolysis requires good knowledge of the kinetics reaction. The complexity of the enzymatic hydrolysis of PALF come from the fact that they are heterogeneous insoluble substrate, and thus, their enzymatic hydrolysis was always limited. Henri-Michaelis-Menten equation was agreed by most researcher as a kinetic enzyme that can only use for homogeneous system (Chrastil and Wilson, 1988; Chrastil, 1988). However, Baley *et al.* (1989) proposed an alternative approach to the classical Henri-Michaelis-Menten equation suggests that the initial velocity, v_o, should be expressed as a function of the initial enzyme concentration, E_o, instead of initial substrate concentration, S_o (Baley *et al.,* 1989). This alternative approach has been done by Carillo *et al.* (2005), Pratto *et al.* (2016) and Carvalho *et al.* (2013) to study the kinetics reaction of enzymatic hydrolysis on wheat straw, sugarcane straw and sugarcane bagasse, respectively. On the other hand, study about kinetic coefficients determination of enzymatic hydrolysis of PALF have not been explored yet, therefore, throughout this study, kinetics of the enzymatic hydrolysis of PALF on heterogenous system was done.

1.2 Problem Statement

Pineapple leaves fiber (PALF) are one of the abundant biofiber that have a highest cellulose and hemicellulose content compared to other biofiber and other agricultural waste. However, the main problem in this study is the difficulty to produce reducing sugar from PALF, as they have recalcitrant structures and the expensive process in hydrolytic enzyme. The arrangement of PALF are complex because of its fiber arrangement. Daud *et al.* (2014) reported that PALF contained bundles of packed fiber on the surface area where it filled with many fiber matrix that were condensely packed together, thus make it harder to remove lignin and disrupt the lignin-carbohydrate complex (Daud *et al.,* 2014; Reddy and Yang, 2005).

Hence, investigating a suitable pretreatment was a main focus and considered as a key factor for efficient enzymatic hydrolysis process.

There are many pretreatment methods that can be applied to obtained highest yield of reducing sugar. To the best of knowledge, there was only one similar study has been reported on the pretreatment of PALF that was reported by Cherian *et al.,* 2010. The study reported about the isolation of cellulose from PALF by using steam explosion pretreatment. However, the different pretreatment method for PALF and the optimization for the operating condition of the pretreatment have not been explored thoroughly. Therefore, throughout this study, different pretreatment methods was done to investigate the ideal pretreatment method for PALF and the operating condition of ideal pretreatment was optimized to obtained optimum reducing sugar yield.

In addition, optimization of operating condition for enzymatic hydrolysis process was also important to obtain the optimum reducing sugar yield by operate under optimum condition. There were few parameters that need to be considered to optimize the reducing sugar yield included substrate concentration, enzyme loading, temperature and hydrolysis time. Optimization of enzymatic hydrolysis process were important since different pretreatment have different effect on various substrate (Liqian, 2011). Thus, different structure of substrate through pretreatment process cause uncertainty of range for enzyme loading, substrate concentration, temperature and hydrolysis time in hydrolysis process. Moreover, lack of information regarding utilization of PALF in hydrolysis process was the main factor that leads into this study.

1.3 Objectives of Research

1. To determine the best pretreatment method for optimum yield of reducing sugar extracted from PALF.

2. To optimize the operating condition of the best pretreatment method and enzymatic hydrolysis for optimum yield of reducing sugar extracted from PALF.

3. To determine the enzymatic kinetic coefficients of pretreated PALF by heterogenous catalytic equations.

1.4 Scope of Study

To achieve the objectives of this study, several scoped were outlined:

1) Investigation of optimum reducing sugar yield extracted from PALF using different kind of pretreatment which included dilute acid, alkaline and autohydrolysis pretreatment. The best pretreatment method with the highest reducing sugar yield was chosen to be subjected for optimization process.

2) Optimization of operating conditions, using BBD, for ideal pretreatment from scope (1) and enzymatic hydrolysis to obtained optimum yield of reducing sugar extracted from PALF. Optimization of pretreatment condition includes chemical concentration, temperature and pretreatment time while optimization of enzymatic hydrolysis conditions include substrate concentration, enzyme loading, temperature and hydrolysis time.

3) Determination of kinetic coefficients (V_{emax} and K_e) and parameter (n and k) of enzymatic hydrolysis of cellulose for pretreated PALF using heterogenous catalytic equation (i.e modified Henri-Michaelis-Menten and Chrastil approach)

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