OPTIMIZATION OF CASCADE LOW-PRESSURE STEAM HEATING AND ORGANIC ACID PRE-TREATMENT ON PINEAPPLE WASTES FOR FERMENTABLE SUGARS PRODUCTION

NORHAFIZA BINTI NORDIN

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Faculty of Engineering
Universiti Teknologi Malaysia

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DEDICATION

Special dedicated to

My beloved father and mother
Nordin bin Othman
Haslina binti Hussein

My beloved husband
Mohammad Amir Zharif bin Mohamad Azilan

My Siblings
Norhana binti Nordin
Norhaida binti Nordin
Norhazwani binti Nordin
Muhammad Azammuddin Nordin
Muhammad Azimmuddin Nordin

My fellow friends
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ABSTRACT

Sugar synthesized from lignocellulosic biomass can potentially supplement the increasing demand of various applications in industries. Pre-treatment of lignocellulosic biomass is challenging due to the need to break the rigid and compact structure to produce sugar. In this study, pineapple waste (PW) were subjected to a cascade pre-treatment of (i) low pressure steam heating (LPSH) and (ii) maleic acid (MA), which aims to improve delignification, increase enzyme accessibility to cellulose and hemicellulose in the PW while reducing the production of inhibitors. The best conditions for pressure (kPa), solid loading (% w/v) and time (min) of LPSH were screened. The resulting solid biomass from LPSH were proceeded to subsequent pre-treatment of MA and optimized by response surface methodology-based Box-Behnken design, where the influence of pre-treatment temperature, acid concentration and time were studied. A total of 68 % w/w delignification with high hemicellulose removal (79.5 %) were achieved while 77.6 % cellulose was retained in the solid residue after cascading both pre-treatments. No 5-hydroxymethyl furfural (5-HMF) and acceptable amount of furfural (1.8 g/L) were detected in the hydrolysate by High Performance Liquid Chromatography (HPLC) analysis, with negligible amount of phenolic content (0.01 g/L) was observed. In comparison, the PW pre-treatment with combined LPSH and conventional sulphuric acid (H_2SO_4) produced 3.6 g/L of furfural and 0.4 g/L of 5-HMF, at similar optimized conditions. The pre-treated PW were further characterized by scanning electron microscopy and Fourier transform infrared spectroscopy to elucidate structural morphology and functional group changes. The optimized cascade pre-treatments can provide up to 54.79 % of glucose yield and 69.23 % of xylose yield. Furthermore, 67.87 % reduction of lignin content from cascade pre-treatment can substantially enhance the glucose yield up to 95.76 % and xylose yield up to 99.07 % during enzymatic hydrolysis using the mixture of cellulase and hemicellulose. This study shows that the cascade pre-treatment of LPSH and MA can decompose the lignin structure of the biomass with a negligible amount of inhibitors and enhance the effectiveness of enzymatic treatment.

ABSTRAK

Gula yang disintesis daripada biojisim lignoselulosa boleh digunakan untuk mengatasi keperluan permintaan yang semakin meningkat dalam pelbagai aplikasi di dalam industri. Proses prarawatan biojisim lignoselulosa adalah proses yang mencabar disebabkan keperluan untuk memecahkan struktur yang padat dan kukuh untuk menghasilkan gula. Dalam kajian ini, sisa nanas (PW) telah melalui proses kombinasi prarawatan secara turutan yang telah dioptimumkan iaitu (i) pemanasan stim bertekanan rendah (LSPH) dan asid maleik (MA), bertujuan untuk menambah baik peluncuran lignin, meningkatkan kebolehcapaian enzim ke selulosa dan hemiselulosa di dalam sisa nanas sekaligus mengurangkan pengeluaran perencat. Keadaan yang terbaik untuk tekanan (kPa), pemuatan pepejal (% w/v) dan masa (minit) untuk LPSH telah disaring. Kemudian, hasil biojisim pepejal yang dirawat dengan LPSH telah dilanjutkan ke prarawatan kedua iaitu prarawatan dengan MA dan proses ini dioptimumkan dengan kaedah permukaan tindakbalas berdasarkan reka bentuk Box-Behnken, di mana pengaruh suhu prarawatan, kepekatan asid dan masa telah dikaji. Sebanyak 68 %w/w delignifikasi dengan penyingkiran hemiselulosa yang tinggi (79.5 %) telah dicapai sementara 77.6 % selulosa dikekalkan dalam sisa pepejal PW, setelah melalui kedua-dua proses prarawatan. Tiada kandungan 5-*hidroksimetil* furfural (5-HMF) dan sedikit kandungan furfural (1.8 g/L) yang dikesan dalam hidrolisat melalui analisis kromatografi cecair prestasi tinggi (HPLC), manakala jumlah kandungan fenolik (0.01 g/L) boleh diabaikan. Secara perbandingan, prarawatan PW dengan gabungan LPSH dan asid sulfurik konvensional (H_2SO_4) telah berjaya menghasilkan 3.6 g/L furfural dan 0.4 g/L 5-HMF pada keadaan optimum yang sama. PW yang sudah dirawat selanjutnya dicirikan oleh mikroskopi elektron imbasan dan spektroskopi inframerah transformasi Fourier untuk menentukan perubahan morfologi struktur dan kumpulan berfungsi. Kombinasi prarawatan secara turutan yang telah dioptimumkan boleh menghasilkan sehingga 54.79 % glukosa dan 69.23 % xilosa. Tambahan pula, pengurangan 67.87 % kandungan lignin daripada pra-rawatan ini boleh meningkatkan hasil glukosa dengan ketara sehingga 95.76 % dan hasil xilosa sehingga 99.07% semasa hidrolisis enzimatik yang menggunakan campuran selulosa dan hemiselulosa. Kajian ini menunjukkan bahawa prarawatan secara turutan ini boleh menguraikan struktur lignin biojisim dengan jumlah perencat yang boleh diabaikan serta meningkatkan keberkesanan rawatan enzimatik.

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LIST OF SYMBOLS

% - Percentage
∆ - Absorbance
℃ - Degree Celcius
%w/w - Weight per weight
%v/w - Volume per weight
kPa - Pressure
U/g - Units per gram
U/mL - Units per millilitre
M - Molar
mM - Millimolar
min - Minutes
hr - Hour
mL - Millilitre
rpm - Revolution per min
µL - Microlitre
µm - Micrometre
g - Gram
µmole - Micromole
β - Beta
mg/L - Milligram per litre
mg/mL Milligram per millilitre
g/L - Gram per litre
A - Acid Concentration
B - Temperature
C - Time
FPU - Filter Paper Units
U - Units
W - Watt

LIST OF ABBREVIATION

FFTC - Food and Fertilizer Technology Centre
FAO - Food and Agriculture Organization
LPSH - Low-Pressure Steam Heating
MA - Maleic Acid
AA - Acetic Acid
CA - Citric Acid
PW - Pineapple waste
AIL - Acid Insoluble Lignin
DF - Dilution Factor
OFAT - One -Factor-at-a-Time
RSM - Response Surface Methodology
ANOVA - Analysis of Variance
3D - 3 Dimensional
BBD - Box-Behnken Design
FTIR - Fourier Transform Infrared Spectroscopy
SEM - Scanning Electron Microscope
5-HMF - 5-Hydroxylmetyl Furfural
SE - Steam Explosion
pH - Potential of hydrogen
m - slope
DNS - 3,5-dinitrosalicylic acid
UV-Vis - Ultraviolet–visible
ND - Not determined

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CHAPTER 1

INTRODUCTION

1.1 Research Background

According to the Food and Agriculture Organization (FAO, 2021), Malaysia is one of the most important producers of canned pineapples in Asian countries in 2017 and recorded a staggering 299912 tonnes of pineapple crops produced in 2019. Pineapple (*Ananas Comosus*) also ranks the 4th among the fruit crops exported from Malaysia after watermelon, banana and papaya (FFTC, 2019), which plays an important role for the country's socio-economic development either domestic or export market.

PW produced from industrial processing and cultivation comprises of crown, peel and core of the fruit and about 30 – 50% of the total fruit weight is discarded after juice extraction. There are practices for sustainable development of pineapple in Malaysia have been documented in publications such as application for downstream value-added utilization of biomass and waste for further industrial processes like fermentation of sugar (Clauser et al., 2021). The high carbohydrate content in the pineapple waste (PW) makes it a good source for fermentable sugars supply. Extremely significant applications of sugars make it worth to synthesize sugars from biomass-derived resources for the application in fine chemicals (Jarosz, 2015), in replacing petroleum-derived chemicals that used in pharmaceuticals and cosmetics industries (Bhaumik & Dhepe, 2015; Wang et al., 2019) and also to the agriculture industries to increase the crop quality and yield (Dotaniya et al., 2016). The highly efficient conversion of lignocellulose to fermentable sugars usually comprises of two major series of biochemical reactions which is pre-treatment and enzyme hydrolysis (Choi et al., 2019). However, the main contributors to the recalcitrance of biomass such as the cellulose crystallinity, the structural heterogeneity, and high lignin content must be overcome to reduce the resistance of the lignocellulosic biomass from sugar hydrolysis (Baruah et al., 2018). To reduce biomass recalcitrance and increase enzyme accessibility to cellulose, pre-treatment that disrupts the biomass cell walls is necessary.

The first series of biochemical reaction which is the pre-treatment is typically required to break down the protective sheath surrounding the lignocellulose so that it becomes suitable to enzymatic hydrolysis which further enhance the accessibility to cellulose and hemicellulose. Numbers of pre-treatment methods have been used by several researchers and most of them are not environmental friendly due to large amount of chemicals used during the process (Baruah et al., 2018; Kumari & Singh, 2018). Consequently, cascade processes that combine acid, alkaline, hydrothermal and/or other pre-treatments have been suggested to maximize sugar recovery and give higher efficiency in lignocellulose digestibility compared to one-step pre-treatment (Moodley & Kana, 2017; Sun et al., 2016; Tang et al., 2020). Guo et al. (2013b) observed a significantly higher sugar recovery (glucose (>80 %) and xylose (>70 %)) from dried *Miscanthus* obtained and by-products formation were remarkably reduced after cascading the low severity acid with alkaline pre-treatment compared to single stage pre-treatment. This probably because of the different structural features of the biomass wastes (Tang et al., 2020). The level of glucose in the enzymatic hydrolysate pre-treated cornstalk with two-step hydrothermal processes, dilute acid pre-treatment and oxidated alkali pre-treatment at such mild hydrolysis were reached 57.0% , the internal pore volume and porosity also enhanced which created more active accessibilities to enzymes in the residues compared to the single pre-treatment process (Xia et al., 2021).

Method using physiochemical pre-treatment alone such as steam explosion (Zhiqiang et al., 2015), ammonia fiber explosion (Lee et al., 2010), liquid hot water (LHW) (Zhuang et al., 2016), and superheat steam (SS) (Barchyn & Cenkowski, 2014) have been suggested to minimize formation of inhibitory compounds, reduced energy usage without the use of any chemical catalyst, and result in increasing fermentative sugar. Due to the combination of heat and pressure, it prompts the substrate for further breakdown as it is possible to open the bundles of lignocelluloses and make enzymatic reactions easily accessible to the polymer chain of cellulose and hemicellulose (Baruah et al., 2018). Steam pre-treatment without the explosion by using equipment like pressurized vessel (pressure cooker) can be effective in cellulose accessibility as steam with explosion. However to give the essentially the same yields of glucose and total reducing sugars, the treatment must be with saturated steam at high temperature (>240 ℃) (Brownell & Saddler, 1987). Due to the cellulose and hemicellulose structure having different thermal stabilities and endurance, enough pressure should be applied to keep the water in liquid state within the appropriate time, so that the lignin will be degraded the most while the hemicellulose and cellulose remain intact (Cao et al., 2012b). High temperature (220-260℃) are too harsh for the pre-treatment of biomass as low glucose yield was obtained from the supernatant despite the large amount of cellulose and hemicellulose being solubilized and eventually degraded (Weil et al., 1998).

Recently, increasing attention has been paid to acid pre-treatment with respect to industrial implementation, because of their efficiency in not only maximisation of the sugar yields , but also the minimisation of the production costs (Bensah & Mensah, 2013; Carregari Polachini, 2019). However, it gives some drawbacks such as equipment corrosion, gypsum formation during neutralization, sugar degradation due to the high reactivity and formation of inhibitory by-products (Kumar & Sharma, 2017). Compared to alkaline pre-treatment, acid pre-treatment released higher amount of sugar (Baadhe, 2014). Organic acids such as maleic acid, citric acid, formic acid, oxalic acid, acetic etc. which are defined as organosolv system were often applied at higher concentration and their ability has been proven to replace the inorganic acid. It is less toxic and more efficient in carrying out the hydrolysis over a range of pHs and temperatures (Lee & Jeffries, 2011), resulting in minimal xylose and glucose lose because of its superior selectivity to be able to mimic the structure of the specific active sites of enzyme which help to reduce the degradation of glucose (Guo et al., 2013a; Lu & Mosier, 2008; Mosier et al., 2002b) and produce lesser amount of inhibitors (Deng et al., 2016).

Lastly, the second series of biochemical reaction is enzymatic hydrolysis which totally depends on the biocatalysts capability to convert biopolymers into monomers (Wu et al., 2021). Majority of the results obtained from different studies have proved that the combinations of cellulase and hemicellulase enzymes for the hydrolysis of pre-treated lignocellulosic biomass successfully produced higher reducing sugar yield compared to single cellulase or hemicellulase hydrolysis (Amani et al., 2018; Conesa et al., 2016). These two types of enzymes need to work in synergy at optimized hydrolysis conditions to effectively break apart the complex biomass structure and releasing high sugar yield.

1.2 Problem Statement

Many research have been conducted to increase the effectiveness of pre-treatment methods for maximal fermentable sugars production from the lignocellulosic biomass due to the recalcitrant structure. However, efficiency of the single pre-treatment process was often not satisfactory compared to cascade pre-treatment (Kumar & Sharma, 2017).

Pre-treatment with pressure cooker consumed less energy (< 143.27 kPa and <110°C compared to steam explosion (SE) (170°–210°C) and autoclaving method (121°C) (Roda et al., 2016b). SE and autoclaving method usually use pressure above 15 psi and that will increase glucose significantly during the enzyme hydrolysis Unfortunately, xylose were decreased if the pressure used is too high which likely due to solubilization of hemicellulose during the hot water phase (Barchyn & Cenkowski, 2014). Amorphous hemicellulose is known as thermosensitive hemicellulose, where it is sensitive to extreme temperatures (>150°C) and longer residence times, easily degrade 5-carbon xylose sugar into their inhibitory by-products compared to cellulose and lignin (Shaoni Sun et al., 2014) and the hydrolysate need extra detoxification steps after the hydrothermal pre-treatment. The use of water, heat and vapor leads to hemicellulose degradation and lignin transformation owing to the high temperatures approximately 110–300°C. During biomass hydrolysis, hot water cleaves hemiacetal links, releasing acids and facilitating the breaking of ether bonds in biomass (Maneeintr et al., 2018). Higher severity conditions caused accumulation of acid insoluble material that led to recondensation reaction of Klason lignin causing high molecular weight lignin (Kim et al., 2014). These might inhibit the accessibility of cellulose to the enzymatic hydrolysis. In this study, due to the low lignin content of the PW, low pressure steam heating of pressure cooker were used for the 1st stage pre-treatment and optimized with the aim to achieve high delignification but still able to retain high hemicellulose and cellulose content by maintaining the liquid phase (under pressure). The valve regulates the pressure inside the pressure cooker to a pre-set level: typically ~10 psi above atmospheric pressure were able to boil water up to 114°C.

There are studies that investigate the effectiveness of organic acids such as maleic acid, citric acid, acetic acid, oxalic acid and etc. as alternative to the conventional sulphuric acid in order to delignify the lignocellulosic biomass, enhance cellulose and hemicellulose digestibility while reducing the amount of toxic by-products such as furfural and 5-HMF (Cao et al., 2012a; Kootstra et al., 2009a; Sahu & Pramanik, 2018b). By using harsh chemicals from the conventional methods, it required extra detoxification steps due to the production of high total furans (>4.5 g/L) and phenolic content which can also contributed to some sugar losses (Khedkar et al., 2018b). Due to the slightly expensive cost of organic acid pre-treatment compared to inorganic acid, it is suggested to cascade the LSPH with organic acid pre-treatment to reduce the cost.

Hence, this project is focusing on assessing the efficiency of cascade pre-treatment of low-pressure steam heating (LPSH) using simple and economical-value (Roda et al., 2016a) equipment such as commercial pressure cooker (LPSH) and organic acid as means of pre-treating a mixture of whole pineapple wastes parts. The operating conditions of LPSH combined with several organics acids pre-treatment were optimized and characterized together with further optimization of enzymatic hydrolysis conditions for efficient saccharification.

1.3 Objectives of the Study

In general, this work is aimed to improve the pre-treatment process's performance for the conversion of pineapple waste (PW) into fermentable sugars by introducing the cascade of low-pressure steam heating (LSPH) and organic acid pre-treatment. It can be further sub-divided into the following specific objectives:

i. To optimize the LSPH pre-treatment parameters on pineapple waste with the highest lignin removal while retaining the most hemicellulose and cellulose.

ii. To optimize the 2nd stage organic acid pre-treatment parameters of the LPSH-pre-treated pineapple waste for optimum hemicellulose degradation with negligible yield of inhibitors.

iii. To determine the best enzyme mixture ratio for optimum sugar yields and evaluate physical and chemical characterization of the cascade pre-treated PW.

1.4 Scope of the Study

The following are the scopes of the study in order to achieve the objectives:

a) The biomass used in this study consist of the mixed part of pineapple waste such as their leaves, peels, crown, core, and other non-fruit parts with a fixed particle size at 500 µm.

b) The parameters chosen for the optimization of the 1st stage pre-treatment (LPSH) are:
    i) Biomass loading (0.5, 1.5, 2.5, 3.5, and 5 % w/v)
    ii) Pressure (40,50, 60, 70 and 80 kPa)
    iii) Time (15, 30, 45, 60 and 75 min)

c) For the 2nd stage pre-treatment, selection of the best organic acids (maleic acid, citric acid, and acetic acid) and solid loadings (%v/w) were carried out based on one-factor-at-a-time 'OFAT' analysis based on the highest xylose and glucose yields released. The other three pre-treatment parameters (acid concentration, treatment time and reaction temperature) were statistically optimized using 3-level-Box-Behnken design (BBD) matrix in the Response Surface Methodology (RSM), Design Expert Software (version11.0.) (State-Ease, USA). The ranges of the three parameters are shown below:

   i) Acid concentration (0.1-1.0 M)
   ii) Treatment time (30-120 min)
   iii) Reaction temperature (50-180°C)

d) The yield of 5-Hydroxymethyl furfural (g/L) and furfural (g/L) inhibitors were analyzed using High Performance Liquid Chromatography (HPLC), while phenolic content (g/L) were analyzed using total phenolic content assay.

e) The efficiency of the pre-treatments were characterized by using Scanning Electron Microscope (SEM) for morphological analysis, Fourier Transform Infrared Spectroscopy (FTIR) for the functional group analysis, Glucose and Xylose Assay Kits for the determination of xylose and glucose yield and DNS method for reducing sugar yield.

f) Screening and optimization of enzyme mixture (cellulase and hemicellulose) hydrolysis of the pre-treated PW were carried out using OFAT with the target of achieving maximum sugar yield. The studied parameters were:

   i) Solid loading (5%, 10%, 15% w/v)
   ii) Time (2, 4, 6, 8, 24 hr)
   iii) Single enzyme loading of cellulase (2, 2.5, 3, 4 FPU/mL) and hemicellulose (4, 12, 20, 40, 60, 80 U/mL)
   iv) Enzyme mixture ratio (1:1, 1:2, 2:1, 3:1, 1:3)

1.5 Significance of the Study

Overall, this study could significantly provide explanation on ecological alternative to reduce fruit processing waste from being directly discarded to the environment. Since detail investigation on the relationship between parameters and aspect ratio will be done in this research, it will provide good mechanism on the technique on producing high yield of reducing sugar with the optimum reaction during hydrolysis of sugar. The process sequence that will be developed from this study can be potentially adapted onto larger scale for further study. The pineapple biorefinery can be used as a source for edible sugar production which can be employed by the nutraceutical and functional food industries and thus may give economic benefit to the whole community.

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LIST OF PUBLICATION

Indexed Conference Proceedings

1. Nordin, N., Illias, R. M., Manas, N. H. A., Nor, A., Ramli, M., & Azelee, N. I. W. (2020, December). Efficient Delignification of Pineapple Waste by Low-Pressure Steam Heating Pre-Treatment. In *Third International Conference on Separation Technology 2020 (ICoST 2020)* (pp. 10-16). Atlantis Press. https://doi.org/10.2991/aer.k.201229.002. **(Indexed by SCOPUS)**

Indexed Journal

1. Azelee, N. I. W., Nordin, N., Illias, R. M., Hasmaliana, N., Manas, A., & Ghazali, M. N. F. M. Enzyme Kinetics Study for Heterogeneous System of Pretreated Kenaf Hydrolysis **(Indexed by ISI)**