UNIVERSITI PUTRA MALAYSIA

HYDROLYSIS OF COTTON FABRIC FROM HOSPITAL WASTE FOR ETHANOL PRODUCTION USING SUB-CRITICAL WATER

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By

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Chair : Nordin Bin HJ Sabli, PhD
Faculty : Engineering

Currently in Malaysia, most wastes are disposed into poorly managed systems with little or no pollution protection measures. Large amounts of wastes such as textiles are generated through hospitals and health care centers. However, the improper management of these abundantly generated wastes may pose an environmental pollution problems and fire hazard. Cotton textile is a potential biomass for bioethanol production. Subcritical water (Sub-CW) hydrolysis was investigated as an alternative technology for the recycling of cotton textile waste for current health care waste management. The aim of this study is to investigate the possibility of complete conversion of cotton textile waste to ethanol via Sub-CW hydrolysis and fermentation. Sub-CW was carried out to facilitate the hydrolysis of cellulose component in cotton textile (cotton 75%+polyester 25%). The study was divided into two parts; (i) To evaluate the subcritical water parameters such as temperature and time to achieve maximum yield of sugars. (ii) Fermentation of the hydrolysate obtained from Sub-CW hydrolysis to ethanol. Under Sub-CW conditions of temperature (140 °C - 350 °C), reaction time (1-10 min) and water to cotton ratio (3:1) revealed that cotton textile treated at 280 °C for 4 min, was optimal for maximizing yield of sugar, which was 0.213 g/g-dry sample. The quantitative analysis by HPLC showed that the soluble carbohydrates in the water phase were mainly composed of glucose. The obtained glucose concentration, 702 mg/L was then fermented at 36 °C for 24 hours by *Saccharomyces cerevisae* (yeast) to ethanol. Maximum yield of ethanol production was 0.105 g/g glucose, which was 17.7% of theoretical yield. Hydrolysis with Sub-CW showed the potential to decompose the cotton textile into simple sugar while keeping sugar degradation to minimal phase and the possibility of complete conversion of cotton textile waste to ethanol via Sub-CW and fermentation.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

HIDROLISIS FABRIK KAPAS DARIPADA SISA HOSPITAL UNTUK PENGHASILAN ETHANOL MENGGUNAKAN AIR SUB-KRITIKAL

Oleh

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Kini di Malaysia, kebanyakan hasil buangan dibuang ke dalam sistem yang tidak terurus dengan sedikit atau tidak ada langkah perlindungan pencemaran. Sebilangan besar buangan seperti tekstil dihasilkan melalui hospital dan pusat penjagaan kesihatan. Walau bagaimanapun, pengurusan yang tidak betul dalam sisa-sisa buangan ini boleh menyebabkan masalah pencemaran alam sekitar dan bahaya kebakaran. Tekstil kapas adalah biomas yang berpotensi untuk pengeluaran bioethanol. Rawatan Subkritikal (Sub-CW) disiasat sebagai teknologi alternatif untuk mengitar semula sisa tekstil kapas untuk pengurusan sisa penjagaan kesihatan semasa. Tujuan kajian ini adalah untuk mengkaji kemungkinan penukaran bahan sisa tekstil kapas kepada etanol melalui Sub-CW hidrolisis dan penapaian. Sub-CW telah dijalankan untuk memudahkan hidrolisis komponen selulosa dalam tekstil kapas (kapas 75% +poliester 25%). Untuk meningkatkan penukaran hidrolisis sisa kapas, parameter air subkritikal dioptimumkan. Kajian ini dibahagikan kepada dua bahagian; (i) Penilaian hydrolysis air subkritikal bagi pelbagai parameter seperti suhu dan masa untuk mencapai hasil maksimum gula (ii) Ketelusan penapaian hidrolisis yang diperoleh daripada air subkritikal. Di bawah keadaan rawatan suhu Sub-CW (140 °C - 350 °C), masa reaksi (1-10 min) dan nisbah air kepada kapas (3:1) mendedahkan bahawa tekstil kapas dirawat pada 280 °C selama 4 minit, adalah optimum untuk memaksimumkan hasil gula, iaitu 0.213 g/g-kering sampel. Analisis kuantitatif oleh HPLC menunjukkan bahawa karbohidrat larut dalam fasa air terutamanya terdiri daripada glukosa, Kepekatan glukosa yang di hasilkan iaitu 702 mg/L kemudian ditapai pada 36 °C selama 24 jam oleh yis Saccharomyces cerevisae kepada etanol. Hasil pengeluaran etanol maksimum adalah 0.105 g/g glukosa, yang merupakan 17.7% hasil teoritikal. Hidrolisis dengan Sub-CW menunjukkan potensi untuk mengurai tekstil kapas kepada gula mudah sambil mengekalkan degradasi gula ke fasa minimum dan kemungkinan penukaran lengkap sisa tekstil kapas kepada etanol melalui Sub-CW dan penapaian.

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This thesis was submitted to the Senate of University Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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- The research conducted and the writing of this thesis was under our supervision
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LIST OF ABBREVIATIONS

Sub-CW	Subcritical water
%	Percentages
^{o}C	Degree Celsius
~	Roughly equal to
<	Less than
>	More than
Conc	Concentration
CO2	Carbon Dioxide
g	Gram
HPLC	High Performance Liquid Chromatography
Kw	Ionization constant
mg	Milligram
MPa	Mega Pascal
P	Pressure
T	Temperature
TOC	Total Organic Carbon
TS	Total sugar
IC	Inorganic carbon
TC	Total carbon
TS	Total Solid

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Serious problem due to depletion of crude oil, economic, and reducing of greenhouse gas emission from fossil fuels have led to the extensive exploration of renewable energy sources. In order to reduce the dependence on non-renewable resources, a full recycling of agro-industrial lignocellulosic wastes such as textile must take place (Canabarro et al., 2017; Liguori & Faraco, 2016; Timung et al., 2015; Vani et al., 2012). Cotton textile is the main supply of natural fiber, hence, this renewable feedstock which is a significant potential for production of different biofuels have been used to produce valuable products such as bioethanol (Wanassi, Azzouz, & Hassen, 2016). Textile waste is a mixture of fibres such as cotton, polyester, wool, silk, nylon, etc., where the first two are the most commonly used due to high energy content (Ryu, Phan, Sharifi, & Swithenbank, 2007). Cotton (Gossypium hirsutumx) is one of the most abundant crops is a significant source for the textile industry (Mcintosh, Vancov, Palmer, & Morris, 2014).

Medical health care waste management has to play an important role to deal with consumption and disposal of solid waste such as cotton fabric. Approximately 12, 000 tons of cotton linen were consumed and 54 tons were disposed annually in hospitals of 3 selected countries in Malaysia (Medilaund Sdn. Bhd.). The treatment and disposal of hospital waste is a subject of concern and controversy to the public and become a major challenge for many governments. The average amount of municipal solid waste daily generation in Malaysia was 29,711 tons/day in 2012 and it is assumed to increase about 36,165 tons/day by the year 2020 (Rezaei, Ghobadian, Samadi, & Karimi, 2018). In Kuala Lumpur, waste generation is about 3000 tons/day and around RM 1billion is needed to dispose of these wastes (R. P. Singh, Singh, Araujo, Hakimi Ibrahim, & Sulaiman, 2011).

According to Ministry of Health Malaysia (2009), hospital waste also includes general waste (Omar, Nazli, & Karuppannan, 2012) and it constitutes about 80% of the total health care waste fraction (Minoglou & Komilis, 2018). General waste which includes a large part of hospital waste has a resemblance to municipal solid waste and is possible to be disposed of in controlled landfills (Mosquera, Andrés-Prado, Rodríguez-Caravaca, Latasa, & Mosquera, 2014). The main method to treat Municipal solid waste (MSW) in Malaysia is landfill (93.5%) where only 5.5% is recycled and only 1.0% is composted.

The current textile recycling practices emphasize on using textiles as feedstock for energy production through combustion or chemical reactions (Yousef,

Tatariants, et al., 2019). Knowledge regarding the quality of disposed textiles is very limited, whether it can be reused, recycled or treated as waste (Nørup, Pihl, Damgaard, & Scheutz, 2018). Cotton or cellulose based waste can be converted into energy through chemical, thermal, and thermochemical approaches but there are limitations like yield, economic profit, time, chemicals consumed, etc. (Yousef, Eimontas, et al., 2019).

Health hazards such as communicable diseases, bad odors, and environmental effects are the complications of inefficient solid waste management (Verma, Borongan, & Memon, 2016). Basic infrastructure, regulations, policies, and efficient MSW managements are what most Southeast Asian Cities lack (Verma et al., 2016). System of door-to-door purchasing of recyclables in Malaysia did not succeed because the local authorities' knowledge and skills related to cycling are not sufficient (Mongkolnchaiarunya, 2005). Recycling is an expensive process and its facilities are not easily accessible to low income communities (Mazibuko et al., 2019).

1.2 Problem Statement

Similar to lignocelluloses, waste textiles have a cellulosic part that can be converted to bioethanol (Shen et al., 2013a).. However, higher crystallinity of cellulose and well-organized construction of the textiles of polyester and cotton fibers from the blending process also will limit the efficiency of cellulose hydrolysis to fermentable sugar (Gholamzad et al., 2014; Jeihanipour & Taherzadeh, 2009; Khaleghian, Karimi, & Behzad, 2015). Conversion of this abundant and renewable biomass into ethanol needs an economically feasible process (Daylan & Ciliz, 2016; Du et al., 2016; Gonçalves et al., 2014).

Various concerns in the healthcare field arose as a result of outdated and inappropriate waste disposal techniques (Thakur & Ramesh, 2015).. Currently in Malaysia the major fraction of health care waste collected is disposed directly in landfills (general waste) (R. P. Singh et al., 2011). Traditional landfill method is linked to problems like pollution and land shortage (H. Zhou, Long, Meng, Li, & Zhang, 2015). Leachate production at poorly maintained landfill sites can result in soil and groundwater contamination (Afroz, Hassan, & Ibrahim, 2003). Incineration is an alternative to landfill, thus pollute the environment during incineration (Aishah, Abd, Yin, Rosli, & Chen, 2013; Gholamzad et al., 2014). Poor management of waste will cause loss of energy and resources which could instead be recycled (Demirbas, 2011).

There is a need to develop effective approaches as the current ones are not fully developed (H. Shi, Liu, Li, & Xu, 2017). Other technologies are more adoptable, because the waste generated has high organic and moisture content (Kathirvale, Noor, Yunus, Sopian, & Halim, 2003). Recently a lot of material and energy recovery technologies have been created (Shekdar, 2009). Waste to energy (WtE) has been acknowledged as an alternative to overcome waste generation

problem and as a potential renewable energy source (S. Tan, Hashim, Lee, Taib, & Yan, 2014), for example incineration, pyrolysis, gasification and anaerobic digestion (Dastjerdi, Strezov, Kumar, & Behnia, 2019). Under WtE idea, cotton textile waste can be converted into a lot of things such as biogas, bioethanol and electricity (Rizwan, Saif, Almansoori, & Elkamel, 2018). However the use of these methods produces toxic substances such as dioxin and furans as well as mercury (Mohseni-Bandpei et al., 2019). In addition WtE technology is considered as the least effective process for waste treatment and energy recovery (Dastjerdi et al., 2019) and the technology is more expensive compared to energy sources such as fossil fuels, natural gases, windpower and hydropower (Maryam Mohammadi & Harjunkoski, 2020).

To overcome these issues, subcritical water (sub-CW) hydrolysis is considered. Sub-CW is used as an environmental friendly method to decompose hazardous matters due to its extraordinary properties (Kirmizakis, Tsamoutsoglou, Kayan, & Kalderis, 2014). Subcritical water also has been proposed as an alternative way for conversion of biomass and cellulose hydrolysis (Abdelmoez, Nage, Bastawess, Ihab, & Yoshida, 2014a). It refers to water at temperature between 100 and 374 °C and at a pressure which is high enough to maintain the liquid state (below the critical pressure of 22 MPa). No chemical reagents used (Huang et al., 2017), inexpensive, non-flammable, non-toxic and non-explosive (Hata, Wiboonsirikul, Maeda, Kimura, & Adachi, 2008). Another advantage of this method is problems like acid waste and corrosion of equipment can be avoided, only water is necessary without addition of any chemicals (Lü & Saka, 2010).It produces higher quality of the extracts with minimal degradation of sugar (Prado, Lachos-perez, Forster-carneiro, & Rostagno, 2015). For raw sample, the process does not require a drying step since water is use as a solvent (Latawiec, Swindell, & Reid, 2008).

Subcritical water (sub-CW) hydrolysis and extraction of valuable compounds from many different biomass has been actively studied (Lachos-Perez et al., 2017) recently. They indicate that sub-CW can be highly efficient in the process of breaking the lignocellulosic structure of biomass into fermentable sugars within a few minutes compared to other technologies such as enzymatic hydrolysis and does not require pretreatment (Abaide, Mortari, et al., 2019a). For instance, (S. Xu et al., 2021) have studied the subcritical water extraction of cotton flowers to obtain bioactive compounds at 180 ^{o}C, the authors obtained isoquercetrin, with its content of 110.54 mg/g. Prado et al. (Prado, Follegatti-Romero, et al., 2014) observed the sugarcane bagasse treated with subcritical water hydrolysis showed the presence of 5.6% of monosaccharides, cellobiose and cellotriose at 213 ^{o}C and 33 ml/min in fermentable sugars. Besides Abdelmoez et al. (Abdelmoez, et al., 2014) evaluated the sub-CW hydrolysis effect on wheat straw, the highest yield of total reducing sugar was 51.5% of the raw wheat straw at 190 ^{o}C and 30 min hydrolysis time. Subcritical water hydrolysis done by (Prado, Forster-Carneiro, et al., 2014) from coconut husk, defatted grape seed and pressed palm fiber where experiments were performed at different temperatures ranging from 208 °C to 257 °C for 30 min at 20 MPa showed maximum yields of 30–40% of total sugars in the raw material. Zhao et al. (Zhao, Lu, Wang, & Yang, 2009) reported the highest fermentable hexoses

yield at very short contact times of 27.4% (280 °C, 27 s) and 6.7% (280 °C, 54 s) from corn stalks and wheat straw respectively using sub-CW and supercritical water reactions. Abaide et al. (Abaide, Ugalde, et al., 2019a) obtained fermentable sugars yield of 18.0 ± 2.9 g/100 g dry rice husks by sub-CW hydrolysis in a semi-continuous mode at 220 °C. Pourali et al. (Pourali, Asghari, & Yoshida, 2010) have studied the hydrolysis and decomposition of rice bran using sub-CW to obtain phenolic compounds and valuable materials at temperature range from 100 to 360 ◦C for 10 min with a batch type reactor and produced 215 glucose equivalents mg/g dry matter. However, Oliveira et al. (Oliveira et al., 2020) have investigated the sub-CW hydrolysis of sugarcane bagasse, the highest sugar yield of 3.0 ± 0.53 g L^{-1} xylose and 6.3 ± 0.5 g L^{-1} glucose were achieved at the optimal conditions of 200 ◦C, 15 MPa, 20 e 5 mL min^{-1} .

A survey of literature on cotton textile reported on various sugar extraction, hydrolysis methods and ethanol fermentation. To the best of our knowledge there was a gap of knowledge in the area. No research has been carried out on ethanol production especially using sub-CW hydrolysis of cotton textile as pre-treatment to produce sugars especially for the purpose of fermentation process. Thus, the aims of this work are to investigate the possibility of sub-CW on cotton-based waste textiles for sugar recovery and ethanol production by using yeast strain of *Saccharomyces cerevisiae* in fermentation. The variables considered were reaction temperature and time.

1.3 Research Questions

1. How effective of Subcritical water (sub-CW) treatment in hydrolyzing the cellulose content of cotton-based waste textiles to produce high quality of extract?

2. Does the temperature, time applied and properties of the sample influence the efficiency of the treatment?

1.4 Objectives

The aim of this study is to investigate the possibility of complete convertion of cotton textile waste (Cotton 75% & polyester 25%) from hospital to ethanol via sub-CW and fermentation. To achieve the aim mentioned above, the following technical objectives have to be made.

1) To evaluate the Sub-CW experimental condition in order to obtain highest yield of sugar by varying reaction temperature and time.

2) To investigate the possibility of converting obtained reducing sugar to ethanol via fermentation after sub-CW by comparing with commercialized sugar (white sugar).

1.5 Scope of Study

This research work covers the application of sub-CW hydrolysis of cotton textile (cotton 75%+polyester 25%) from cotton textile waste to sugars (monosaccharides) and fermentation of obtained reducing sugar to ethanol. The study is divided into three main stages 1) Characterization of cotton textile which include measurement of moisture content to get the dry weight of sample, ash content and chemical composition. 2) Investigation of different parameters that affect hydrolysis process. Two parameters were manipulated when doing Sub-CW hydrolysis, which were temperature (140 – 350 °C) and time (1 - 10 min) of hydrolysis. Size of the sample loaded in the reactor was not manipulated and kept constant for all experiments as it does not give significant effect on the hydrolysis process. 3) Several analyses were performed on the collected hydrolysate which include total organic compounds (TOC), phenol-sulphuric acid method for total sugar content and high performance liquid chromatography (HPLC) to quantify sugars produced. Solid residues produced were also collected and weighed.

The analysis of reaction temperature and time was conducted based on the highest yield of total sugar. The hydrolysate from sub-CW hydrolysis was further fermented with baker yeast, Saccharomyces cerevisae (S. cerevisae) to obtain highest yield of ethanol via fermentation process. Meanwhile the results were compared with white sugar as reference material. The sample reading for ethanol was taken hourly for 24 hours. Sugar and ethanol profile was quantified using High Performance Liquid Chromatograph (HPLC). This study highlights the potential of hydrolysis through sub-CW to produce sugars and ethanol for recycling of cotton textile waste.

1.6 Significant of the Study

Sub-CW can offer many opportunities, to promote successful long term solutions to the problem faced by economic and environmental. The decomposition process is equivalent to autoclave sterilization process, it can provide sterilization far superior than autoclave and the system can be installed within hospital. It can turn the infectious waste into harmless general waste to be discarded. This system not only minimize the solid waste volume to be disposed into landfills, but reduce the solid waste biodegradable to zero and at the same time generates valuable biofuel products for recycling. This significantly reduces the disposal costs and the need for additional landfills across the country. The recycle cotton textiles make it possible to increase yields of ethanol compared to other raw lignocelluloses due to high cellulose content. CO_2 emission can be

reduced far lower than the incinerator and no dioxin emission, while giving consideration to the environment. In addition, this process is very simple to implements and this results in a significant phase-out of technologies that are redundant such as collection of mixed waste, waste incineration and land filling.

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