



UNIVERSITI PUTRA MALAYSIA

***PRODUCTION OF SUGARS FROM SAGO BARK USING SUBCRITICAL
WATER TREATMENT***

NURHUSNI BINTI M. AMIN

FK 2020 88



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WATER TREATMENT**

By

NURHUSNI BINTI M. AMIN

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in
Fulfilment of the Requirements for the Degree of
Master of Science**

December 2019

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

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NURHUSNI BINTI M. AMIN

December 2019

Chair : Nordin Bin Hj. Sabli, PhD
Faculty : Engineering

Sago bark is one of the solid wastes being discarded in large volume from sago industries and currently being abandoned and underutilized. The effort to valorize this waste is not enlarge due to the technology limitation. Besides, the current practices on the sago wastes left at the sago starch processing industries have raised to various environmental issues. Due to its large in carbon content, sago bark is subjected to subcritical water treatment in this study for producing sugars and other value-added products. Sago bark was differentiated into inner and outer layers and both were treated at 180 - 320 °C for 5 min. It was found that inner layer yielded higher amount of TOC (0.27 g/g-dry sago bark) and total sugar (0.67 g/g-dry sago bark) than the outer layer at 210 °C. Inner layer was also substantially affected with more than 90% solid loss recorded with increasing reaction temperature and maximum tar yield of 0.41 g/g-dry sago bark at $T \geq 280$ °C. Besides, inner layer had shown better conversion and solubility in subcritical water thus, it is more susceptible to subcritical water treatment than the outer layer. Inner layer was selected for further experiments under varied reaction times, 1 - 15 min. Temperatures of 180, 210 and 240 °C were selected and made constant at a time. Temperature of 210 °C and reaction time 5 min yielded the highest total sugar yield among the other reaction temperatures. The production of tar was less affected by the prolonged reaction time unlike at higher temperature. It was noticed that polysaccharide in the bark decomposed sequentially from higher to lower degree of polymerization. Hemicellulose decomposed earlier (180 °C, 5 min) than the cellulose portion (≥ 210 °C, ≥ 7 min). Among all of reaction temperatures applied, 240 °C showed better monosaccharides yields (glucose = 0.093, xylose = 0.097 g/g-dry sago bark) in shorter time, 5 min. 210 °C was a suitable temperature for arabinose production with the highest yield of 0.015 g/g-dry sago bark at 2 min. The drop of pH combined with increase amount of organic acids generated confirmed that, sugars degradation had happened at higher reaction temperature and time. The six types of organic acid identified were pyruvic, malic, acetic, formic, succinic and lactic acids. The declining thermogravimetric analysis combustion values, from 80 to 60 wt. % for the reaction temperature 180 - 240 °C further supported that sago bark had successfully decomposed during the reaction. The thermogravimetric analysis also revealed that hemicellulose decomposed the most during the reaction rather than the

cellulose and resulted in large contribution to the sugar yields. The increasing crystallinity index obtained, 59.3 - 78.1% when treated at 180 - 240 °C as compared to the raw sago bark, 38.6%, further verified the efficient removal of certain amount of lignin and hemicellulose from the solid sample matrix. The crystallinity index became evident that only amorphous region of cellulose was affected thus, contributed into the cellulose sugars production i.e. glucose at higher temperature and time.



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

PENGHASILAN GULA DARIPADA KULIT KAYU SAGU MENGGUNAKAN RAWATAN SUBKRITIKAL

Oleh

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Kulit kayu sagu adalah salah satu sisa pepejal yang dibuang dalam jumlah yang besar dari industri sagu dan kini diabaikan dan kurang digunakan. Usaha untuk mempelbagaikan kegunaan sisa ini tidak diperbesar kerana keterbatasan teknologi. Selain itu, amalan meninggalkan buangan sisa sagu di kilang-kilang telah membawa kepada pelbagai isu alam sekitar. Disebabkan kandungan karbonnya yang banyak, kulit kayu sagu telah dirawat dengan air subkritikal dalam kajian ini untuk penghasilan gula dan produk nilai tambah yang lain. Kulit kayu sago dibezakan kepada lapisan dalam dan luar dan kedua-duanya dirawat pada suhu 180 - 320 °C selama 5 minit. Lapisan dalam menghasilkan jumlah TOC (0.27 g/g-kulit sagu kering) dan jumlah gula yang tinggi (0.67g/g-kulit sagu kering) berbanding lapisan luar pada suhu 210 °C. Lapisan dalam juga merekodkan kehilangan pepejal melebihi 90% apabila suhu tindak balas meningkat dan menghasilkan tar maksimum sebanyak 0.41 g/g-kulit sagu kering pada suhu ≥ 280 °C. Selain itu, lapisan dalam telah menunjukkan keterlarutan yang lebih baik dalam air subkritikal dan lebih mudah terurai dalam air subkritikal berbanding lapisan luar. Lapisan dalam telah dipilih untuk tindak balas seterusnya pada masa tindak balas yang berbeza, 1 - 15 minit. Suhu 210 °C dan masa tindak balas 5 minit telah menghasilkan jumlah gula tertinggi di antara suhu-suhu yang lain. Penghasilan tar kurang dipengaruhi oleh masa tindak balas yang lama tidak seperti pada suhu yang tinggi. Polisakarida dalam kulit kayu terurai secara berurutan dari tahap pepolimeran yang tinggi kepada tahap yang rendah. Hemiselulosa diuraikan lebih awal (180 °C, 5 min) berbanding selulosa (≥ 210 °C, ≥ 7 min). Di antara suhu tindak balas yang digunakan, 240 °C menunjukkan hasil monosakarida yang lebih baik (glukosa = 0.093, xylosa = 0.097 g/g-kulit sagu kering) dalam masa yang lebih singkat, 5 minit. 210 °C adalah suhu yang sesuai untuk pengeluaran arabinosa dengan hasil tertinggi sebanyak 0.015 g/g-kulit sagu selama 2 minit. Penurunan pH dan peningkatan jumlah asid organik mengesahkan bahawa kerosotan gula berlaku pada suhu dan masa tindak balas yang lebih tinggi. Enam jenis asid organik telah dikenalpasti iaitu asid piruvik, malik, asetik, formik, susinik dan laktik. Nilai pembakaran analisis termogravimetrik yang menurun dari 80 hingga 60 wt. % bagi suhu 180 - 240 °C, menyokong bahawa kulit kayu sagu telah berjaya diuraikan semasa tindak balas. Analisis termogravimetrik juga mendedahkan bahawa hemiselulosa lebih terurai semasa rawatan berbanding selulosa dan banyak menyumbangkan kepada

penghasilan gula. Peningkatan indeks kristaliti yang diperolehi, 59.3 - 78.1% apabila dirawat pada 180 - 240 ° C berbanding dengan kulit kayu sagu mentah, 38.6%, selanjutnya mengesahkan penyingkiran efisien sejumlah lignin dan hemiselulosa dari matriks pepejal sampel. Indeks kristaliti menjadi bukti bahawa hanya selulosa rantau amorf yang terjejas dan telah menyumbang kepada penghasilan gula selulosa seperti glukosa pada suhu dan masa yang lebih tinggi.



ACKNOWLEDGEMENT

I express my deepest grateful to Allah S.W.T for His guidance and strength that enabled me to complete this master study.

I would like to thank my supervisor and my supervisory committee Dr. Nordin Hj. Sabli and Ir. Dr. Shamsul Izhar Siajam for their advices, guidance and constructive criticisms throughout my master journey. Special thanks to Prof. Hiroyuki Yoshida who had initiated this topic and research area from the beginning of this study. I would also want to express my sincere thanks to laboratory members and staffs, for their support and encouragement and their willingness to help me through when in need.

Most importantly, I am also greatly indebted to my beloved ibu and ayah and all family members for their endless prayers, support and understanding while completing my study. Not to forget my dear husband, Nur Taufiq Bin Jamalludin who never failed to keep me accompany and support me emotionally through the ups and downs.

Thank you also to Universiti Putra Malaysia for the research funding and the financial aids provided to support my study.

I really appreciate all the contributions received throughout my study years and this master journey would be the bittersweet memory that I will keep forever in my heart.

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

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LIST OF ABBREVIATIONS

SCW	Subcritical water
SB	Sago bark
TOC	Total organic carbon
TGA	Thermogravimetric analysis
TS	Total sugar
XRD	X-ray Diffraction
AS	Acetone soluble
WS	Water soluble
RS	Residual solid



CHAPTER 1

INTRODUCTION

1.1 Background of Study

Sago palm is a starch crop par excellence, as it has a higher starch production capacity, between 20 - 25 tons/ha/year, than that of cassava, rice or corn (Flores, 2008). The world production of starch is approximately about 27.5 million tons. Meanwhile, global consumption of sago starch accounts for about 3% of the total world market which lies between 200,000 to 300,000 tons per annum and the rest is dominated by corn, potato and tapioca (Bujang, 2006). Even though current staple food has been diverged to rice, there are still numerous products derived from sago for it has multitude usage. For instance in food industry, sago is widely used to produce sago pearls, sago flour and syrups in soft drinks. Besides, it is also used in the glue industry involved in plywood manufacture (Singhal et al., 2008).

In Malaysia, there are more than 90% of all sago-planting areas concentrated in the state of Sarawak, which is one of the world's largest sago exporter, exporting about 25,000 - 40,000 tons of sago products annually to Peninsular Malaysia, Japan, Taiwan, Singapore, etc. (Bujang, 2006; Singhal et al., 2008). In the world, Malaysia is currently the third main sago producer after Indonesia and Papua New Guinea and combined, the three countries produce approximately 94.6% of the world's sago (Naim et al., 2016). Although Indonesia and Papua New Guinea produce more sago than Malaysia, the sago industry in this country is well established and has become one of the important industries contributing to export revenue (Karim et al., 2008). In Malaysia, sago starch ranks fifth in terms of highest agricultural revenue after pepper, palm oil, cocoa, and rubber (Abd-Aziz, 2002). Department of Agricultural Sarawak, DAS reported that the production and export value of sago starch has been increasing 15% to 20% every year (Jenol et al., 2014; Mohamad et al., 2011). As the demand increases, so will sago production, which consequently contributes to the significant amount of waste generated.

In the sago starch processing industries, there are three by-products generated namely sago bark, SB fibrous pith residue or also known as *hampas* and wastewater. Bark and *hampas* are classified as solid residues whereas wastewater is a liquid residue. In a day, about 237.6 tons of wastewater and 7.1 tons of fibrous *hampas* are generated from approximately 600 logs of sago palm (Zainab et al., 2014). Meanwhile, about 5 -15 tons SB is produced from the processed logs each day from a factory (Vikineswary et al., 1994). This huge value eventually amounts to more than 20, 000 tons of sago bark being discarded annually from Malaysia's sago industry.

Currently, the SB wastes left behind during the starch processing are destroyed through open or control burning, discarded into the rivers or left to degrade naturally at the

factories. These practices pose several serious problems such as threat to the environment through air pollution, shallow the rivers and create unpleasant odor and view (Wahi et al., 2014). Nonetheless, initiatives have been taken by locals when using SB wastes to build a platform around the factory, footpaths of houses, wall materials, and fences. Besides, there are also numerous interior decoration products from SB such as wall tiles, furniture and flower pots recycled from SB in ascribing its natural features and beautiful surface (Abd Rahman, 2005).

Subcritical water (SCW) is a condition when water maintains its liquid form between water boiling temperatures, 100 °C to its critical temperature, 374 °C under adequate pressure. In the last decades, SCW treatment has gained increased attention due to several advantages over other biomass extraction methods including the ability to use wet heterogeneous biomass feedstock without prior dewatering (Pavloviè et al., 2013b). Also, there are several factors that can influence the efficiency of SCW treatment such as reaction temperature, time, pH and etc. Nonetheless, reaction temperature gives a substantial influence on SCW reaction than the other factors.

1.2 Problem Statement

There is an abundant amount of SB being discarded yearly from sago starch processing industries and are not fully utilized. The current practices on the sago wastes left at the sago starch processing industries have raised to an environmental issue as it is loaded in the fields to naturally degrade or practically burnt. Exploitation of SB wastes are not maximize and only minimal efforts have been done either industrially or in research institutions (Abd Rahman, 2005) that rooted from no clear new technology can be adapted for alternative sago bark utilization (Chong et al., 2014). More extensive research on SB should be done pertinent to its large content of cellulose and hemicellulose compositions. According to Ethaib et al. (2016), SB has high in carbohydrate content, accounting for more than 60% of cellulose and hemicellulose. Therefore, it can be used to produce sugars as a feedstock or raw materials in other industries i.e. biofuel industry. This could enhance the usage of SB instead of utilize it for traditional building materials.

Recently, several researches have successfully been done on SB in producing xylitol (Mohamad et al., 2013) via acid hydrolysis and fermentation, and sugars (Ethaib et al., 2016) using microwave-assisted dilute acid pretreatment and enzymatic hydrolysis. Both studies have resulted in a very promising value. Nonetheless, the applications of chemicals and enzymes during reactions are of apathy since awareness towards green solvent and environmental friendly becomes the major concerns. Application of acid is corrosive and required neutralization process while enzymes require very long time to obtain the products and cannot be recycled. Hence, in this research a method known as subcritical water (SCW) treatment is introduced to address the aforementioned problem while maximizing the SB utilization.

SCW treatment used water at high temperature between 100 to 374 °C under an adequate pressure of ~22.0 MPa. However, the high temperature treatment posed in this process

may rapidly degrade the products produced, thus, several key parameters should be manipulated and controlled to ensure the maximum product yields are achieved. Besides, the presence of lignin that bound closely around the cellulose and hemicellulose components makes the hydrolysis accessible restricted. This is more prominent at the outer bark where lignin is mostly concentrated over 20% at this region than the inner bark (Pásztor et al., 2016). Therefore, distinguishing and separating the bark layers could enhance the accessibility of hydrolysis process and sugars production. Most of the sugars production from biomass wastes could be contributed from either cellulose or hemicellulose as the major carbohydrate portions in plants. Solid characterization such as TGA and XRD analyses (Ethaib et al., 2016; Mohan et al., 2015) on remaining solid left after hydrolysis may tell the either cellulose or hemicellulose reacted the most and contributed to product yields.

1.3 Objectives

In this study, SB was proposed as the raw material for the production of valuable materials using SCW to enhance the utilization of sago wastes. Therefore, the objectives of this study are as follows:

1. To compare the conversion between inner and outer layers of SB using SCW treatment into any value-added products.
2. To evaluate the effects of SCW treatment process conditions (reaction temperature and time) on SB in producing sugars and other value-added products.
3. To characterize the residual solid left from SB after SCW treatment.

1.4 Scope of Research

To conduct the first objective, the SB was debarked manually and differentiated into inner and outer layers, concerning that the different distance of layers from the center pith could influence the sago trunk anatomically and starch content. Then the samples were chopped into smaller sizes, 2 - 3 cm according to the specified SB wastes usually discarded from sago starch industries. The layers were characterize in terms of moisture and ash content as well as its chemical compositions. Then, the different SB layers were subjected to SCW treatment at varied temperatures, 180 to 320 °C and constant time, 5 min. 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 of a batch system (Prado et al., 2015). Meanwhile, hydrolysis using water at room temperature was used as a control. Three phases were isolated after treatment called WS, AS phases and residual solid. TOC and TS analyses were conducted on WS phase to ensure the decomposition of SB and the presence of sugar after treatment respectively. While, gravimetric analysis done on AS phase and residual solid left to monitor the possible effects of pyrolysis that may take place at high reaction temperature. Solubility of the two layers in SCW treatment was compared, and the layer with high solubility indicates high reactivity and reliability of the material.

For the second objective, the selected layer that produced high TS yield was further reacted with SCW treatment. Three best reaction temperatures were fixed separately to

constant at a time under varied reaction time, 1 to 15 min. Similar analyses were done as in previous (in objective 1), with additional HPLC-sugar and HPLC-organic acid analysis to identify the type of sugar and organic acid present as a function of time and temperature respectively. The significance analysis, $p < 0.05$ of reaction temperature and time and its interaction was conducted using two-way ANOVA analysis based on the TS sugar yield. Tar and residual solid collected was limited to only study on its yield.

For the third objective, solid characterization analysis was done in terms of thermal stability and crystallinity of solid residue using thermogravimetric and X-ray diffraction methods respectively. The remaining solid residue left after SCW treatment at 180, 210 and 240 °C and 10 min was categorized as treated SB and analyzed to compare its solid structure with the untreated SB. Solid characterization analysis would give ideas on the extensive effects of SCW on SB and the chemical composition of SB that was affected and contributed to the product yields.

1.5 Significant of The Study

SCW treatment on SB will definitely provide the environmental friendly alternative solution for issues related with sago starch processing industries. Production of valuable materials from SB by fully utilizing the cellulosic materials present will enhance the zero emission from biomass waste and/or agriculture waste in Malaysia. It will reduce wastes being dump into landfills as well as the burning practice that consequently cause increment in the carbon footprint. The production of value-added products such as sugar from SB will harness the generation of bio-based fuel i.e. bioethanol, biobutanol and etc. Thus, this research will positively affect our society, nation and economy.

1.6 Thesis Outline

This thesis comprised of five chapters. First chapter briefly describes the background of the study, problem statement, objectives and outline of the thesis. The second chapter reviews in details about the sago palm, current status of sago wastes and its utilization, the various hydrolysis techniques including SCW treatment as well as biomass that had been treated with SCW hydrolysis. Chapter three mainly describes in details about the sample preparation, step of procedures, calculations, chemicals and equipment involved throughout the research. Meanwhile, chapter four reports and elaborates all of the results obtained in regards to the experiments done with justification and support from previous studies. Final chapter, chapter five summarizes all the findings according to the research objectives and recommends some suggestions for improvements as for the continuity of future researches.

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LIST OF PUBLICATIONS

Journal

Amin, N., Sabli, N., Izhar, S., & Yoshida, H. (2020). Production of valuable materials from sago bark using subcritical water treatment. *Int. J. Eng. Res. Technol.*, 13(1), 1-11.

Amin, N., Sabli, N., Izhar, S., & Yoshida, H. (2019). Sago wastes and its applications. *Pertanika J. Sci. & Technol.*, 27(4), 1841-1862.

Conference

1. Nurhusni M. Amin, Nordin Sabli, Shamsul Izhar and Hiroyuki Yoshida (2017). Production of Valuable Materials from Sago Bark Using Subcritical Water Treatment. Oral Presentation. *The Asian Conference on Oleo Science 2017*, 11th - 13th September 2017. Tokyo University of Science, Japan
2. Nurhusni M. Amin, Nordin Sabli, Shamsul Izhar and Hiroyuki Yoshida (2017). Extraction of Sago Bark Using Sub-critical Water Treatment. Oral Presentation. *Wood and Biofiber International Conference (WOBIC2017)*, 21st - 23rd December 2017. Hotel-Bangi Putrajaya, Malaysia



UNIVERSITI PUTRA MALAYSIA

**STATUS CONFIRMATION FOR THESIS / PROJECT REPORT
AND COPYRIGHT**

ACADEMIC SESSION : _____

TITLE OF THESIS / PROJECT REPORT :

**PRODUCTION OF SUGARS FROM SAGO BARK USING SUBCRITICAL
WATER TREATMENT**

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NURHUSNI BINTI M. AMIN

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