UNIVERSITI PUTRA MALAYSIA

XYLOSE PRODUCTION OF OIL PALM FROND USING DILUTE ACID HYDROLYSIS

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By

HADI AMIRKHANI

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

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DEDICATION

I dedicate this thesis to my beloved parents, wife and brothers
for their love, endless support and encouragement
with love…

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

XYLOSE PRODUCTION OF OIL PALM FROND USING DILUTE ACID HYDROLYSIS

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HADI AMIRKHANI

December 2013

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Faculty: Engineering

Oil Palm Frond (OPF) is a lignocellulosic waste from palm oil plantation. This study focuses on the first part of the two-stage dilute acid hydrolysis of OPF fiber namely the conversion of hemicellulose to simple sugar (xylose as a type of pentose sugar).The characterisation of OPF was accomplished by using thermogravimetric analysis (TGA) and fiber analysis to determine cellulose, hemicellulose and lignin. The hydrolysis process was conducted in a batch reactor. The experimental began with acid screening to select the most effective acid for OPF hydrolysis. Sulphuric acid (H_2SO_4), hydrochloric acid (HCl) and acetic acid (CH_3COOH) were chosen as the potential catalysts and the acid hydrolysis was done at 100^oC, solid to liquid ratio of 25:1 (w/v) and 2% (v/v) concentration of acid. Consequently,H_2SO_4 was found to yield the highest amount of xylose. The effect of the solid to liquid ratio (SLR) was then studied using 2% sulphuric acid by varying the ratio from 1:20 to 1:35 (w/v) at100^oC.

The ratio 1:30 gave the highest amount of xylose, hence it was chosen as an optimum solid to liquid ratio for subsequent experiments. The optimization study was conducted on acid hydrolysis of OPF fiber using sulphuric acid at SLR of 1:30. The manipulated reaction conditions were temperatures (100-140^oC), acid concentrations (2-6%) and reaction times (0-240 min). The analysis of three mono-sugars namely xylose (main mono-sugar),glucose and arabinose as well furfural as inhibitor were determined using high performance liquid chromatography (HPLC). Based on the potential amount of xylose (10.8mg/ml), 94% conversion was obtained under the optimum conditions. This optimum yield was achieved at 2% (v/v) acid concentration, at 120^oC in 120 minutes. The possibility of improving the hydrolysis OPF using ultrasound-assisted pre-treatment of OPF fiber was also investigated. The effects of ultrasonic power (40, 60, and 80%of 300 Watt) and irradiation time (20, 30, 40, and 60min) were examined. After ultrasonic pre-treatment, thefibers were subjected to hydrolysis conducted at optimum conditions of hydrolysis process (2% acid sulphuric, 120^oC and 120 minutes). The maximum yield of xylose at 7.31 mg/ml was achieved at 80% power after 40 minutes of ultrasonication. The yield was lower than the hydrolysis without pre-treatment because most of the sugar was degraded to furfural. The amount of furfural rose from 0.0163 mg/ml to 0.063 mg/ml after pre-treatment at optimum condition. The kinetics study on the dilute acid hydrolysis of OPFfiber revealed that the hydrolysis reaction is a first order

irreversible reaction. Dilute acid hydrolysis reaction was analyzed using kinetics models developed by Saeman. Kinetics constants for Saeman model were analyzed using Arrhenius type expansion which includes activation energy and catalyst concentration factors.

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

PENGHASILAN XYLOSE DARIPADA PELEPAH KELAPA SAWIT MELALUI KAEDAH HIDROLISIS ASID CAIR

Oleh

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Pelepah minyak sawit (PMS) adalah sisa lignoselulosa dari ladang kelapa sawit. Kajian ini menumpu kepada bahagian pertama pada dua peringkat hydrolysis asid cair pelepah minyak sawit (PMS) iaitu penukaran hemiselulosa kepada gula ringkas (xylose). Pencirian PMS dilakukan dengan menggunakan analisis Termogravimetri (TGA) dan analisis gentian. Proses hidrolisis telah dijalankan didalam reaktor pilot kelompok. Eksperimen dimulakan dengan penyaringan asid untuk memilih asid yang paling berkesan untuk hidrolisis PMS. Asid sulfurik (H_2SO_4), asid hidroklorik (HCl) dan asid asetik (CH_3COOH) telah dipilih sebagai pemangkin yang berpotensi dan proses hidrolisis asid telah dilakukan pada suhu 100˚C, nisbah pepejal kepada cecair 25:1 (w/v) dan kepekatan asid 2% (v/v). Sebagai hasilnya, H_2SO_4 didapati menghasilkan jumlah xylose yang tertinggi. Kesan nisbah pepejal kepada cecair kemudiannya dikaji menggunakan asid sulfurik 2% dengan mengubah nisbah daripada 1:20 kepada 1:35 (w/v) pada suhu 100˚C.

Nisbah 1:30 telah memberikan jumlah xylose yang tertinggi, maka ia telah dipilih sebagai nisbah pepejal kepada cecair yang optimum untuk eksperimen berikutnya. Kajian pengoptimuman telah dijalankan kepada hidrolisis asid gentian PMS menggunakan asid sulfurik pada nisbah pepejal kepada cecair 1:30. Keadaan tindak balas yang dimanipulasikan adalah suhu (100-140˚C), kepekatan asid (2-6%) dan masa tindak balas (0-240 minit). Analisis tiga gula utama, iaitu glukosa, xylose (terutamanya mono-gula) arabinose dan furfural sebagai perencat telah ditentukan dengan menggunakan kromatografi cecair prestasi tinggi (HPLC). Berdasarkan jumlah potensi xylose (10.8mg/ml), 94% penukaran telah diperolehi di bawah keadaan optimum. Hasil optimum ini dicapai pada kepekatan asid 2% (v/v), pada suhu 120˚C dan dalam masa 120 minit. Kemungkinan meningkatkan hidrolisis PMS menggunakan pra-rawatan ultrabunyi-terbantu juga telah disiasat. Kesan parameter termasuk kuasa ultrasonik (40, 60, dan 80%) dan masa penyinaran (20, 30, 40, dan 60 min) telah diperiksa.Selepas pra-rawatan ultrasonik, hidrolisis telah dijalankan kepada gentian pada keadaan optimum proses hidrolisis (kepekatan asid sulfurik 2%, pada suhu 120˚C dan dalam masa 120 minit). Hasil maksimum penghasilan xylose pada 7.31 mg/ml telah dicapai pada kuasa ultrasonik 80% selepas 40 minit. Penghasilan adalah lebih rendah jika dibandingkan dengan tidak menggunakan pra-rawatan kerana kebanyakan gula telah direndahkan kepada furfural. Jumlah fulfural meningkat daripada 0.0163mg/ml sehingga 0.063mg/ml selepas pra-rawatan pada

keadaan optimum. Kajian kinetik yang dilakukan bagi proses hidrolisis asid cair ini mununjukkan tindak balas hidrolisis ini adalah tindak balas oder pertama tidak berbalik. Tindak balas hidrolisis asid cair telah dianalisa dengan menggunakan model kinetic yang dicadangkan oleh Seaman. Pemalar kinetic bagi model Seaman telah dianalisa menggunakan cara pengembangan Arrhenius termasuk faktor tenaga pengaktifan dan kepekatan pemangkin.

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I certify that a Thesis Examination Committee has met on 27 December 2013 to conduct the final examination of Hadi Amirkhani on his thesis entitled "Xylose Production Of Oil Palm Frond using Dilute Acid Hydrolysis" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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