Diluted Sulphuric Acid Hydrolysis of Destarched Sago Fibre assisted with Selected Pre-treatments for Glucose and Xylose Production

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Abstract

In Sarawak, Malaysia, approximately 7 t of sago fibre waste is produced daily from a single sago starch processing mill and it is currently disposed of either directly into a river nearby or in open spaces. On a dry basis, sago fibre contains 58% starch, 23% cellulose, 9.2% hemicellulose and 4% lignin. Our previous study used the trapped starch of sago fibre as a substrate for producing glucose through an enzymatic hydrolysis process in which the destarched fibre (DSF) remained unused. This study represents an attempt to utilise destarched sago fibre (DSF) as a raw material for glucose and xylose production. The DSF initially underwent a pre-treatment process via dilute sulphuric acid hydrolysis to liberate xylose for which four parameters were studied: the solid-to-liquid ratio (5:100-40:100), the dilute sulphuric acid concentration (0% (v/v) - 9% (v/v)), reaction time (30, 60 and 90 minutes) and the effects of steaming and microwave pre-treatment.

Steaming pre-treatment led to the highest xylose (28.19 ± 0.78 g/L) and glucose (78.63 g/L ± 0.22 g/L) production, for which the dilute sulphuric acid concentration was set at 2% (v/v), reaction time was set at 60 minutes. The solid-to-liquid ratio was 30:100. Overall, this work indicates that the optimal pre-treatment of DSF can yield glucose and xylose which can be used to produce bioethanol and xylitol. The study also suggests that DSF can be an alternative raw material for xylitol production.

Keywords: Destarched sago fibre, dilute sulphuric acid pre-treatment, steaming, microwave, mild hydrothermal, glucose, xylose.

Introduction

Biomass resources from agricultural waste are abundant in Malaysia. This type of waste accounts for more than 70 million t annually. Its volume continuously increases each year due to the rapid development in Malaysia[1]. In Sarawak, the sago palm plantation land in the Mukah division contributes one type of agricultural waste. This state, most notably the Mukah division, contains the largest area of sago palm plantation. Sago fibre, bark and wastewater are all waste products of the sago business. Starch and lignocellulosic substances comprise most of this waste such as lignin, cellulose and hemicellulose. Several studies have utilised sago waste to yield valuable sugar products such as glucose[1,19,20]. However, the literature shows that limited studies have been attempted to use sago fibre to produce xylose.

Various processes can be used to perform the hydrolysis pre-treatment of lignocellulosic waste. Dilute acid is one of the most effective in selectively releasing hemicellulosic sugars (xylose and arabinose), leaving a residue containing the cellulose and lignin fractions almost unaltered[18]. Acid hydrolysis is one of the most commonly used methods due to its simplicity, effectiveness and economic feasibility[24]. It was reported that acid hydrolysis pre-treatment removed nearly 35% of the hemicellulose in corncob[25].

Several studies have recently been conducted using different types of lignocellulosic waste as the substrate, all of which underwent dilute sulphuric acid pre-treatment to produce xylose hydrolysate. The pre-treatment was performed using steaming, the reaction times ranged from 1 hour to 1.5 hours and the dilute sulphuric acid ranged from 1% to 2.5% [6,18,23]. However, studies have not been undertaken using sago waste with these selected parameters.

Microwave heating is used as an efficient way to perform the thermal pre-treatment of biomass. It is an alternative to traditional heating due to its high heating rate and ease of preparation[10]. The structure of cellulose, hemicellulose and lignin can be altered by microwave irradiation. Microwave pre-treatment helps to degrade the hemicellulose and lignin. It disrupts the silicified waxy surface, enhancing the enzymatic sensitivity of reducing sugar[14]. The pre-treatment of switchgrass using a microwave helped to raise the sugar yield by around 58%.

Furthermore, pre-treatment using microwave irradiation produced a 56% increment of glucose yield from rape straw[16]. The main benefit of using microwave irradiation is that it aids in cellulosic disintegration. The main advantages of this method over traditional heating are its high uniformity and selectivity, short process time and lower energy requirements. Previous researchers used six seconds of microwave pre-treatment and six minutes of reaction time to produce 88% glucose and 76.5% xylose yields using sago pith waste[28]. Dilute acid with hydrothermal pre-treatment conducted at relatively low temperatures can optimise the xylose released from the hemicellulose[22]. The pre-treatment of wheat straw which utilised a maximum temperature of 140 °C and pH 1, produced 197 g/kg of xylose.

It was concluded that the pre-treatment of wheat straw under the influence of the pH was a significant factor in glucose and xylose yields[22]. Our research involved examining the selected parameter of dilute hydrolysis to produce a liquor with large fractions of glucose and xylose as well as good fermentability to produce high-value-added products like bioethanol and xylitol. The reactions were performed in a batch reactor under different conditions regarding the solid-to-liquid ratio, sulphuric acid concentration and response time. These were selected based on previous research into acid hydrolysis of other lignocellulosic materials.

Material and Methods

Lignocellulosic Compositional Analysis of DSF: The DSF used in this study was obtained from solid particles of leftover hydrolysed starchy sago fibre[19]. The DSF was then air-dried and ground to pass a one-mm screen before compositional analysis to determine its lignocellulose content. The DSF compositional analysis used neutral detergent fibre (NDF) and acid detergent fibre (ADF)[9].

Dilute Sulphuric Acid Hydrolysis of DSF using Selected Physical Pre-treatments: The xylose and glucose recovery from DSF were determined by studying the effects of the sulphuric acid concentration (v/v: 0%, 2%, 4%, 6% and 9%), acid hydrolysis reaction time (minutes: 30, 60 and 90) and the solid-to-liquid ratio of DSF to acid (w/v: 7:100 10:100, 20:100, 30:100 and 40:100). For each of these parameters, three different physical pre-treatments were adopted in this study: steaming, microwave and hydrothermal.

A) Steaming: The steaming process was performed in a vertical autoclave (Shimadzu, Japan) at a pressure of 1.5 atm and a temperature of 121 ℃ for 30 minutes.

B) Microwave: The microwave pre-treatment was carried out in a domestic microwave with a power rating of 510 W at a frequency of 2450 MHz. The microwave's DSF load was fixed at 7% (w/v) to compare this method with the steaming and hydrothermal pre-treatments using the substrate loading. The pre-treatment used an eight-minute reaction time irradiated at 510 W. Then, 7% (w/v) of DSF was prepared using different dilute sulphuric acid concentrations (v/v): 0%, 2%, 4%, 6% and 9%.

C) Hydrothermal: The reactions were performed at 100 ℃ in a water bath (Wisebath). Fixed DSF concentrations of 7% w/v, different dilute sulphuric acid concentrations (0% (v/v), 2% (v/v), 4% (v/v), 6% (v/v)) and 30-minute reaction time were employed in the hydrolysis process. After the pre-treatment and hydrolysis, the hydrolysate was separated from the undissolved DSF using a muslin cloth before being taken for glucose and xylose analysis using HPLC.

Analytical Method: The experiments were related and the mean values were reported. Necessary samples were taken to ascertain the concentrations of glucose and xylose present in the fermentation broth determined by High-Performance Liquid Chromatography (HPLC) using a Shimadzu Chromatographic System (Shimadzu, Kyoto, Japan) equipped with Shimadzu LC-20AT and Shimadzu RID-10A. Before the analysis, each sample was centrifuged at 10,000 rpm for five minutes, filtered using a 0.2 µm Puradisc filter membrane and maintained at 4ºC before being injected into an Aminex Fermentation Monitoring Column (Aminex HPX-87H column 150 x 7.8 mm, BioRad Laboratories, Inc) maintained at 60 ºC. The mobile phase was 5 mM sulphuric acid with a fixed flow rate of 0.8 mL/min.

Statistical Analyses: The data in this study were statistically analysed using the ANOVA single parameter tool (significance level ≤ 0.05). Tukey-test analyses (MS Excel, 2007) were performed for two-tailed distributions to determine whether the datasets differed significantly. Probabilities ≤ 0.05 were considered significant.

Results and Discussion

Lignocellulosic Compositional Analysis of DSF: Table 1 summarises the lignocellulosic composition of the DSF used as a raw material for this study and the design of raw sago fibre based on the findings of other researchers.

All the values in the table are comparable to those already reported. The sago fibre had 52.0% to 58.0% starch percentages whereas the cellulose, hemicellulose and lignin content were 20.7%, 11.2% and 3.1% respectively. The effectiveness of the extraction procedure used by the sago mills determines the starch content in sago fibre[3]. Based on the reported literature, most studies utilised the cellulose content in the sago fibre to maximise glucose production. However, the hemicellulose fraction had not been investigated.

Table 1
Comparison of the composition of raw sago fibre with different collections of sago fibre with DSF

Sago Fibre Collection	Composition (%)		
	Starch	Cellulose	Hemicellulose
Mukah, Sarawak*	0%	20.72	10.12
Mukah, Sarawak[2]	56.0%	20.70	11.20
Pusa, Sarawak[11]	58.0%	21.00	13.40
Local factory, Sarawak[28]	52.0%	16.00	9.80

* This study.

The hemicellulose fraction can be solubilised through dilute acid pre-treatment to produce sugar-rich hydrolysate.

Dilute Sulphuric Acid Hydrolysis of DSF Assisted with Selected Physical Pre-treatments: Three selected physical pre-treatments (steaming, microwave and hydrothermal) were used in this study to assist in the liberation of glucose and xylose sugars from dilute sulphuric acid hydrolysis of DSF.

Effects of different dilute sulphuric acid concentrations on glucose and xylose recovery from DSF: The initial experimental setup used a fixed substrate load, 7 g of DSF mixed with 100 ml of sulphuric acid (H_2SO_4 99%) at different concentrations (0 v/v, 2 v/v, 4 v/v, 6 v/v and 9 v/v) while the reaction time was set at 30 minutes for the steaming and mild hydrothermal pre-treatments with eight minutes appointed for the microwave pre-treatment. This study examined the effects of different dilute sulphuric acid concentrations on the glucose and xylose recovery from DSF, assisted by three physical pre-treatments.

Steaming-Assisted Hydrolysis: The findings related to the liberation of glucose and xylose from the DSF are shown in figures 1, 2 and 3. Based on the results shown in figure 1, the pre-treatment using dilute sulphuric acid in a 2% (v/v) concentration produced the highest xylose concentration, 6.98± 0.03 g/L, followed by 6% (v/v) acid concentration (6.54± 0.21 g/L), 9% (v/v) acid concentration (7.54 ± 0.21 g/L), 4% (v/v) acid concentration (6.95± 0.06 g/L) and lastly, 0% concentration for which no xylose was detected.

The undetectable xylose concentration justified that at 121 ℃, dilute acid would be needed to solubilise the hemicellulose fraction in the DSF to produce sugars. If lignocellulosic waste is pre-treated with water as the solvent, a higher temperature is required for autohydrolysis to solubilise the hemicellulose fraction[7]. Furthermore, previous studies used a higher temperature while in this experiment, the highest temperature was 121℃. The results indicated that 2% v/v acid efficiently solubilised the hemicellulose for the DSF hydrolysate. Previous findings showed |that 2.5% dilute sulphuric acid was best for maximising extensive hemicellulose removal[5]. 9% increase in acid concentration did not cause a significant change in the xylose concentration.

Based on this finding and statistical analysis, the most suitable concentration for dilute sulphuric acid pre-treatment was found to be 2% v/v, as acid at this concentration produced the highest xylose recovery with a xylose concentration of 6.98± 0.03 g/L.

As shown in figure 1, increasing the dilute sulphuric acid concentration helped to increase glucose production; however, the xylose production showed no significant differences. The highest glucose concentration was observed using 9% v/v. The lowest glucose concentration was obtained without dilute sulphuric acid as 3.29 ± 0.46 g/L. Based on the results shown in figure 1, it was concluded that the dilute sulphuric acid pre-treatment yielded higher concentrations of glucose than xylose because acid hydrolysis released the most glucose from the hemicellulose fraction[8]. The dilute acid pre-treatment also solubilised the cellulose fraction, contributing to the higher glucose concentration[15].

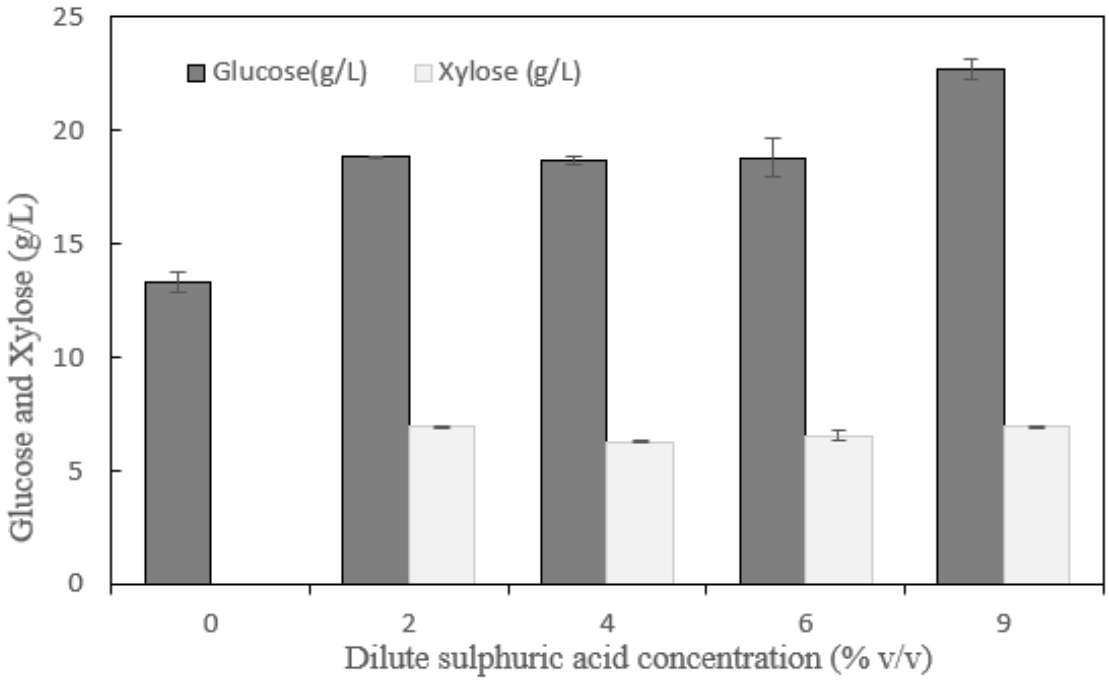

Fig. 1: Glucose and xylose obtained from dilute sulfuric acid hydrolysis of DSF assisted with steaming pretreatment

The p-value from ANOVA was < 0.5, indicating that different acid concentrations improved the xylose and glucose yields. Thus, it was concluded that the optimum level of dilute sulphuric acid to obtain a high xylose concentration was 2 v/v. In comparison, 9 v/v of dilute sulphuric acid was the best option for yielding a high glucose concentration.

Microwave-assisted hydrolysis: The glucose yield obtained from each acid hydrolysis is reported in figure 2. The glucose concentration with this pre-treatment achieved higher results than when the steaming pre-treatment was employed. The highest glucose recovery concentration was 37.73 ± 0.48 g/L using 9% v/v of dilute sulphuric acid followed by concentrations of 6% v/v, 4% v/v, 2% v/v and 0% v/v which produced glucose concentrations of 35.65 ± 0.23 g/L, 31.30 ± 0.08 g/L, 17.76 ± 0.24 g/L and 1.79 ± 0.09 respectively. The glucose concentration levels obtained in these experiments were lower than those obtained in the previous study that utilised sago pith waste[28].

It was reported that 66.5 g/L was produced using 1.0 mol/L H_2SO_4 at 900 W for one minute of microwave heating[28]. One factor contributing to this result was the starch composition of the sago waste. The starch percentage in DSF is 0%, whereas sago pith waste is 52%. However, the DSF could still produce glucose from the cellulose and hemicellulose fractions.

This experiment detected no xylose, even though different acid concentrations were used. The result justifies that the short reaction time of eight minutes was not efficient enough to solubilise the hemicellulose fraction. This result could have been contributed by the microwave-assisted dilute sulphuric acid pre-treatment reaction time of eight minutes at a low voltage of 550 W. It is strongly believed that no hemicellulose degradation occurred. A longer reaction time is needed to yield xylose from DSF hydrolysate. A higher acid concentration and a longer hydrolysis time increased the total sugar and reduced the amount of sugar in the hydrolysate[27].

The ideal hydrolysis conditions for xylose production from lignocellulosic waste such as wheat straw requires a 90-minute reaction time and a 0.5% concentration of sulphuric acid[12]. In contrast, a previous study using sago pith waste reported that increasing the microwave power might positively impact the glucose yield[28]; however, increasing the acid concentration molarity had little effect on the glucose yield.

The p-value from ANOVA was < 0.5, indicating that with microwave pre-treatment, different acid concentrations improved the glucose yield and 9 v/v of dilute sulphuric acid helped to increase glucose production.

Mild Hydrothermal-Assisted Hydrolysis: The hydrothermal pre-treatment was conducted using the same reaction time and solid-to-liquid ratio as the steaming and microwave pre-treatments (30 minutes and 7:100, respectively) but with a different temperature 100 °C.

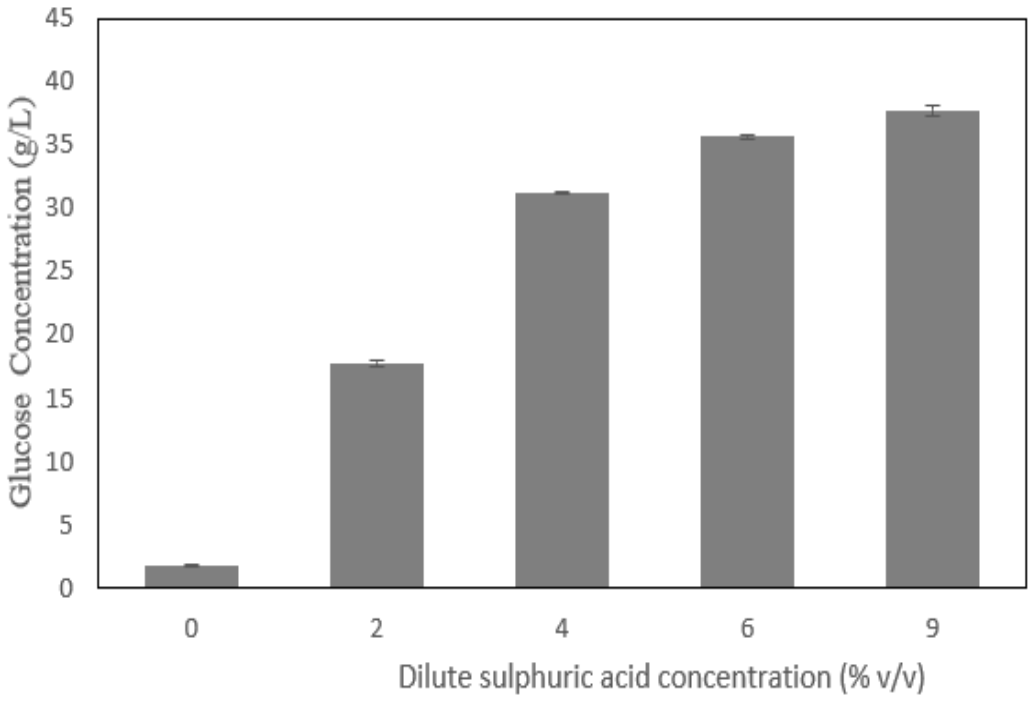

Fig. 2: Glucose obtained from microwave pre-treatment for different dilute sulphuric acid concentrations

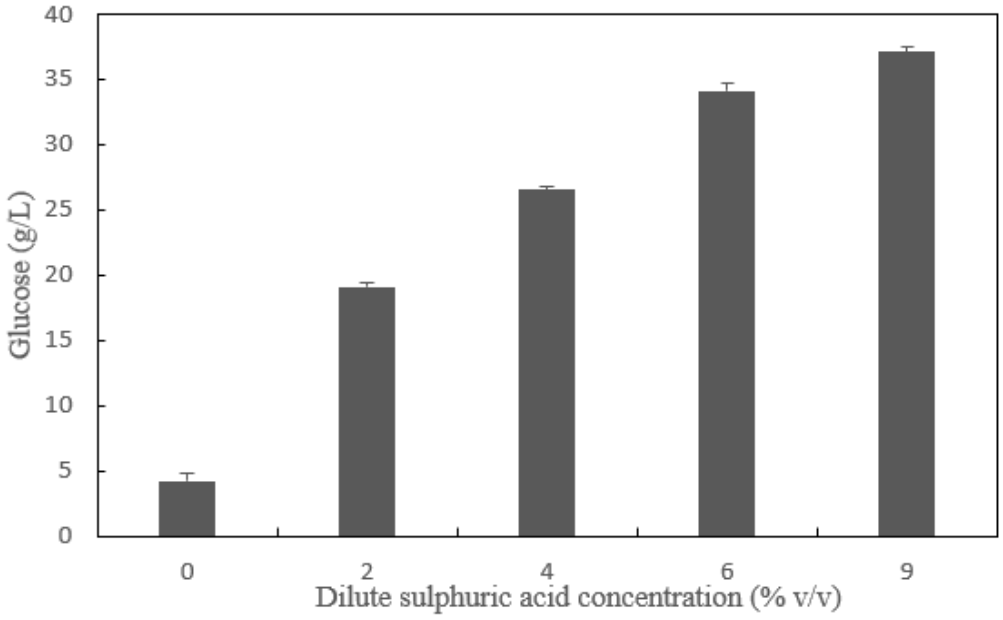

Fig. 3: Glucose (g/L) obtained from mild hydrothermal pre-treatment for different dilute sulphuric acid concentrations (v/v)

Based on the results shown in figure 3, the glucose concentrations obtained from the hydrothermal pre-treatment were 4.20 ± 0.60 g/L, 19.08 ± 0.32 g/L, 26.53 ± 0.23 g/L, 34.13 ± 2.52 g/L and 37.11 ± 1.39 g/L when using acid concentrations of 0, 2, 4, 6 and 9 (v/v) respectively. The highest glucose concentration achieved was when 9% v/v of dilute sulphuric acid concentration was used. Like the microwave pre-treatment, the hydrothermal pre-treatment failed to yield any xylose from the DSF hydrolysate.

The result justifies how at 100 ℃, the hydrothermal pre-treatment was not sufficiently high in temperature to solubilise the hemicellulose fraction to produce xylose. The glucose recovery might have come from the cellulose-glucose conversion in the DSF hydrolysate. This result suggested that the hydrolysis of lignocellulosic at a low temperature required the help of enzymes. This was supported by the previous finding that the hydrolysis of bamboo could be conducted at 50 ℃ with a 60-minute reaction time and with enzymes[26]; this yielded 86% and 82.6% of glucose and xylose respectively[26].

Another possible reason why xylose was not detected, may be the conversion of xylose into another byproduct because a prolonged reaction time with dilute acid hydrolysis could cause a conversion of xylan through the cyclodehydration process into furfural, an inhibitory product of fermentation[17]. This result showed that microwave- and hydrothermal-assisted pre-treatment could not recover xylose sugar from the hemicellulose. One factor contributing to this could be the crystalline structure of the lignocellulosic. The recalcitrance of sago fibre reduces the solubility of the hemicellulose part to break down into sugars such as xylose and other oligosaccharides[21].

This study concluded that steaming pre-treatment using an autoclave would be the best option to yield xylose sugars as this pre-treatment produced the highest xylose concentration using 2% v/v of dilute sulphuric acid. Further parameters such as the reaction time and solid-to-liquid ratio were further investigated through steaming pre-treatment utilising an autoclave. The p-value from ANOVA was < 0.5, indicating that the different acid concentrations with mild hydrothermal pre-treatment improved the glucose yield and 9 v/v of dilute sulphuric acid helped to increase glucose production.

Effects of different hydrolysis reaction times on glucose and xylose recovery of DSF: Only the pre-treatment of DSF using steaming produced xylose sugar. 2% (v/v) sulphuric acid parameter led to a better xylose recovery of 6.98±0.03 g/L than microwave and hydrothermal pre-treatment. Therefore, this study aimed to examine how the hydrolysis reaction time (30, 60 and 90 minutes) affected the glucose and xylose recovery from DSF hydrolysed using 2% (v/v) sulphuric acid and a 7% solid-to-liquid ratio.

As shown in figure 4, the highest xylose yield was achieved at 30 minutes of reaction time as 8.42 ± 0.23 g/L whereas the highest glucose concentration was noticed at 60 minutes as 20.62 ±0.15 g/L. At 90 minutes, both the glucose and xylose concentrations decreased slightly. It was reported that with a longer reaction time, xylose could decompose into furfural[4]. The decomposition of the sugar might cause this due to the prolonged heating time and the sugar might dehydrate into furan compounds[30]. The result also indicated the presence of high levels of acetic acid with concentrations of 17.67 g/L and 21.98 g/L at 60-minute and 90-minute reaction times respectively.

The high acetic acid concentration in the liquid indicated that the sugars were being degraded after their release from the lignocellulosic matrix[4]. Acetic acid is a product of the auto-hydrolysis of the acetyl (–$COCH_3$) group in hemicellulose molecules. These compounds are detrimental to fermentation due to their inhibitory behaviour, causing the medium's pH to decrease[4]. Thus, it was concluded that different reaction times minimally affected both xylose and glucose production. The most suitable reaction time for the pre-treatment to produce the xylose concentration was 30 minutes which produced te highest xylose concentration for xylitol fermentation. A shorter reaction time with a high sugar recovery would be preferred due to the increased glucose production and xylose recovery.

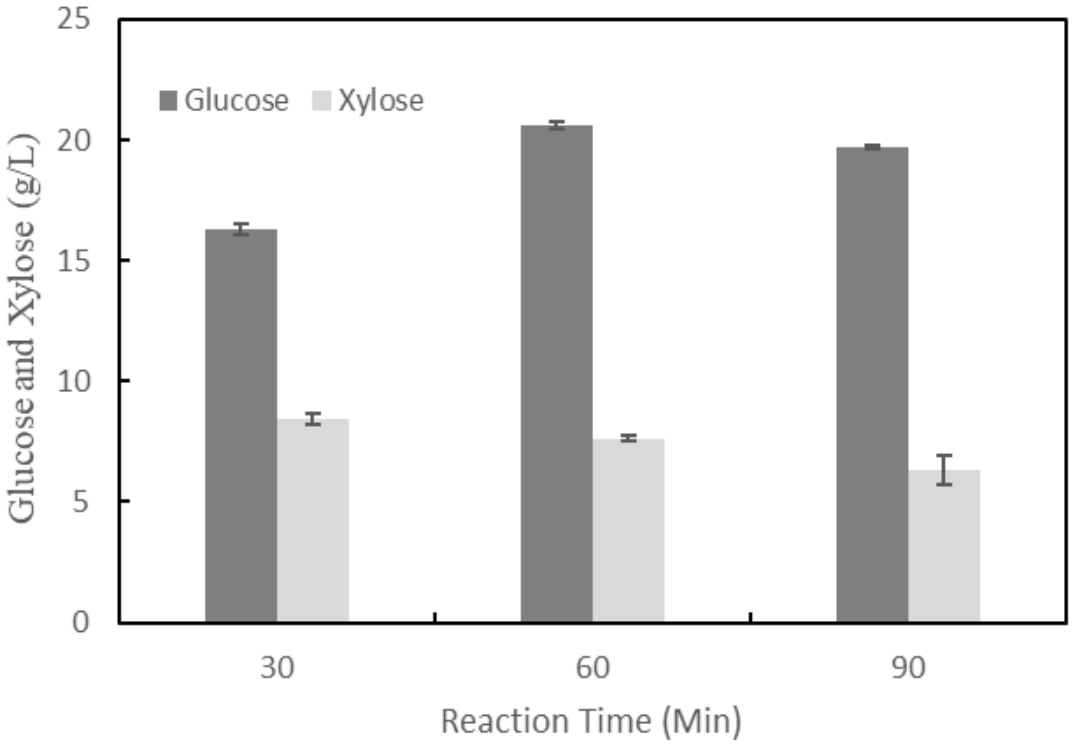

Figure 4: Glucose and xylose (g/L) obtained from steaming pre-treatment for different reaction times

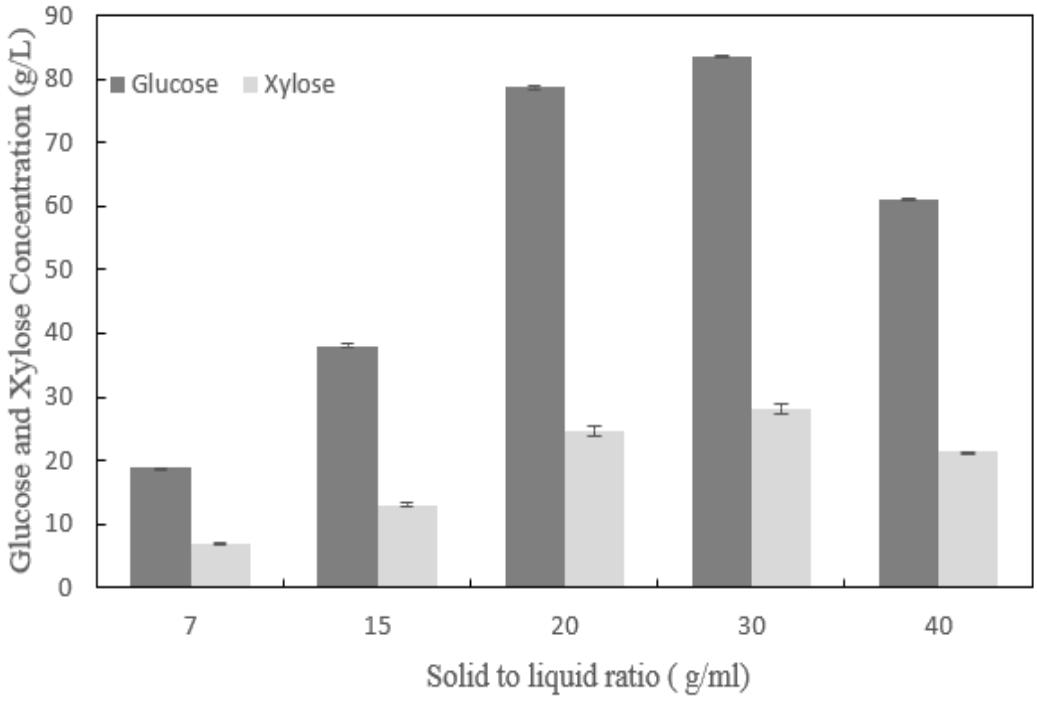

Fig. 5: Glucose and xylose obtained from steaming pre-treatment for different solid-to-liquid ratios

Effects of different solid-to-liquid ratios of DSF to 2% (v/v) sulphuric acid on glucose and xylose recovery: To examine the impact of the substrate load on the glucose and xylose recovery from DSF, four different solid-to-liquid ratios (7:100, 15:100, 20:100 and 30:100) were set up. Based on the previous results, 2% (v/v) sulphuric acid concentration and 30-minute reaction time were used for all the trials. Based on the results shown in figure 5, the glucose concentration consistently increased with the higher solid-to-liquid ratios. The highest level of glucose and xylose as 83.61 ± 0.05 g/L was achieved with a 30:100 solid-to-liquid ratio. At a 40:100 solid-to-liquid ratio, the concentration of glucose and xylose decreased. Thus, a higher solid-to-liquid ratio is not recommended as the solvent will be insufficient to soak the substrate's surface area for acid hydrolysis thoroughly.

Previous studies stated that high substrate concentrations could inhibit substrate, significantly lowering the hydrolysis rate[13]. Additionally, DSF hydrolysate can absorb water like a sponge. This caused the sample to expand and reduced the dilute sulphuric acid solution available to react with the DSF hydrolysate. This reduced the accessibility of the acid attached to the DSF hydrolysate. Thus, the acid could not cover all the DSF hydrolysate surface area for effective hydrolysis. Based on the ANOVA test, the p-value was <0.5 and no significant differences existed. Based on the findings from this study, it was concluded that the best solid-to-liquid ratio for pre-treatment is 20%. The selected parameters could be used as fermentation media to produce value-added products such as bioethanol and xylitol.

Conclusion

DSF is widely known as a promising substrate for yielding glucose and xylose sugar to produce bioethanol and xylitol. It was concluded that steaming pre-treatment using an autoclave helped to increase glucose and xylose production. In this study, the feasibility of the xylose could be improved by implementing pre-treatment parameters such as the dilute sulphuric acid concentration, reaction time, pre-treatment approach and solid-to-liquid ratio. The pre-treatment of DSF assisted with dilute sulphuric acid was discovered to yield the recovery of high xylose and glucose concentrations using a high-pressure steam autoclave.

Overall, 2% v/v dilute sulphuric acid hydrolysis with a 30:100 solid-to-liquid ratio, a 30-minute reaction time and integrated with steaming pre-treatment exhibited the most of xylose (28.19 g/L) and glucose (83.61 g/L). With the same substrate loading of 7% w/v and a 9% v/v concentration of dilute sulphuric acid, the microwave and mild hydrothermal pre-treatment methods were found to be valuable alternatives to conventional heating as the former produced high glucose concentrations of 37.73 g/L and 37.11 g/L respectively.

In comparison, the high-pressure steam pre-treatment produced a concentration of 22.68 g/L. This research suggested that microwave and mild hydrothermal pre-treatments can be used as alternative ways to obtain glucose sugar for ethanol fermentation. In contrast, high-pressure steam could be an alternative method of xylose sugar production for xylitol fermentation. Further fermentation parameters using *Candida tropicalis* yeast could also be studied to increase xylitol production from DSF hydrolysate.

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References

1. Agamuthu P., Challenges and opportunities in agro-waste management: An Asian perspective, In Inaugural meeting of first regional 3R forum in Asia, University of Malaya, 11-12 **(2009)**

2. Alias Nurul Haziqah, Suraini Abd-Aziz, Lai Yee Phang and Mohamad Faizal Ibrahim, Enzymatic saccharification with sequential-substrate feeding and sequential-enzymes loading to enhance fermentable sugar production from sago hampas, *Processes*, **9(3)**, 1-16 **(2021)**

3. Awg-Adeni D.S., Bujang K.B., Hassan M.A. and Abd-Aziz S., Recovery of glucose from residual starch of sago hampas for bioethanol production, *BioMed Res. Int.*, **2013**, 1-8 **(2013)**

4. Batista Gustavo, Renata B.A. Souza, Bruna Pratto, Martha S.R. dos Santos-Rocha and Antonio J.G. Cruz, Effect of severity factor on the hydrothermal pretreatment of sugarcane straw, *Bioresour. Technol*, **275**, 321-327 **(2019)**

5. Canilha L., Santos V.T., Rocha G.J., Almeida e Silva, Giulietti M., Silva S.S., Felipe M.G., Ferraz A., Milagres A.M. and Carvalho W., A study on the pretreatment of a sugarcane bagasse sample with dilute sulfuric acid, *Journal of Industrial Microbiology and Biotechnology*, **38(9)**, 1467-1475 **(2011)**

6. Cheng Ke-Ke, Jian-An Zhang, Hong-Zhi Ling, Wen-Xiang Ping, Wei Huang, Jing-Ping Ge and Jing-Ming Xu, Optimization of pH and acetic acid concentration for bioconversion of hemicellulose from corncobs to xylitol by *Candida tropicalis*, *Biochem. Eng. J.*, **43(2)**, 203-207 **(2009)**

7. Cuevas Manuel, Juan F. Garcia, Gassan Hodaifa and Sebastian Sanchez, Oligosaccharides and sugars production from olive stones by autohydrolysis and enzymatic hydrolysis, *Ind Crops Prod.*, **70**, 100-106 **(2015)**

8. Debiagi Flávia, Tiago Bervelieri Madeira, Suzana Lucy Nixdorf and Suzana Mali, Pretreatment efficiency using autoclave high-pressure steam and ultrasonication in sugar production from liquid hydrolysates and access to the residual solid fractions of wheat bran and oat hulls, *Appl. Biochem. Biotechnol.*, **190**, 166-181 **(2020)**

9. Hong Eunsoo, Jinyeong Kim, Seunggyo Rhie, Suk-Jin Ha, Junghoe Kim and Yeonwoo Ryu, Optimization of dilute sulfuric acid pretreatment of corn stover for enhanced xylose recovery and xylitol production, *Biotechnol. Bioprocess Eng.*, **21**, 612-619 **(2016)**

10. Hu Zhenhu and Zhiyou Wen, Enhancing enzymatic digestibility of switchgrass by microwave-assisted alkali pretreatment, *Biochem. Eng. J.*, **38(3)**, 369-378 **(2008)**

11. Jenol Mohd Azwan, Mohamad Faizal Ibrahim, Ezyana Kamal Bahrin, Seung Wook Kim and Suraini Abd-Aziz, Direct bioelectricity generation from sago hampas by *Clostridium beijerinckii* SR1 using microbial fuel cell, *Molecules*, **24(13)**, 2397 **(2019)**

12. Ji, Xingxiang, Hao Ma, Zhongjian Tian, Gaojin Lyu, Guigan Fang, Jiachuan Chen and Haroon A.M. Saeed, Production of xylose from diluted sulfuric acid hydrolysis of wheat straw, *Bio Resources*, **12(4)**, 7084-7095 **(2017)**

13. Kristensen Jan B., Claus Felby and Henning Jørgensen, Yield-determining factors in high-solids enzymatic hydrolysis of lignocellulose, *Biotechnol. Biofuels*, **2**, 1-10 **(2009)**

14. Kumari D. and Singh R., Pretreatment of lignocellulosic wastes for biofuel production: a critical review, *Renew. Sustain. Energy Rev.*, **90**, 877-891 **(2018)**

15. Lenihan P., Angela Orozco, Eddie O'Neill M.N.M., Rooney Ahmad D.W. and Walker G.M., Dilute acid hydrolysis of lignocellulosic biomass, *Chem. Eng. J.*, **156(2)**, 395-403 **(2010)**

16. Lu Xuebin, Bo Xi, Yimin Zhang and Irini Angelidaki, Microwave pretreatment of rape straw for bioethanol production: focus on energy efficiency, *Bioresour. Technol.*, **102(17)**, 7937-7940 **(2011)**

17. Mamman Ajit Singh, Jong-Min Lee, Yeong-Cheol Kim, In Taek Hwang, No-Joong Park, Young Kyu Hwang, Jong-San Chang and Jin-Soo Hwang, Furfural: Hemicellulose/xylosederived biochemical, *Biofpr.*, **2(5)**, 438-454 **(2008)**

18. Manjarres-Pinzón, Katherine Mario Arias-Zabala, Guillermo Correa-Londoño and Eduardo Rodriguez-Sandoval, Xylose recovery from dilute-acid hydrolysis of oil palm (Elaeis guineensis) empty fruit bunches for xylitol production, *Afr. J. Biotechnol*., **16(41)**, 1997-2008 **(2017)**

19. Mohammad S., Awg-Adeni D.S., Bujang K.B., Vincent M. and Baidurah S., Potentials of sago fibre hydrolysate (SFH) as a sole fermentation media for bioethanol production, IOP Conf. Ser.: Mater. Sci. Eng., **716(1)**, 012001 **(2020)**

20. Muradi Nur Adila, Awang Adeni Dayang Salwani and Nurashikin Suhaili, Enhancement of very high gravity bioethanol production via fed-batch fermentation using sago hampas as a substrate, *As Pac J. Mol. Biol. Biotechnol*., **28(3)**, 44-51 **(2020)**

21. Pang Suh Cem, Lee Ken Voon and Suk Fun Chin, Conversion of sago (Metroxylon sagu) pith waste to fermentable sugars via a facile depolymerization process, *Appl. Biochem. Biotechnol.*, **184**, 1142-1154 **(2018)**

22. Pedersen Mads, Katja S. Johansen and Anne S. Meyer, Low temperature lignocellulose pretreatment: effects and interactions of pretreatment pH are critical for maximizing enzymatic monosaccharide yields from wheat straw, *Biotechnol. Biofuels*, **4**, 1-10 **(2011)**

23. Ping Yuan, Hong-Zhi Ling, Gang Song and Jing-Ping Ge, Xylitol production from non-detoxified corncob hemicellulose acid hydrolysate by Candida tropicalis, *Biochem. Eng. J.*, **75**, 86-91 **(2013)**

24. Rafiqul I.S.M., Sakinah A.M.M. and Karim M.R., Production of xylose from Meranti wood sawdust by dilute acid hydrolysis, *Appl. Biochem*., **174**, 542-555 **(2014)**

25. Rajeev Ravindran and Jaiswal Amit Kumar, A comprehensive review on pre-treatment strategy for lignocellulosic food industry waste: challenges and opportunities, *Bioresour.*, **199**, 92-102 **(2016)**

26. Sathitsuksanoh, Noppadon Zhiguang Zhu, Tsung-Jen Ho, Ming-Der Bai and Yi-Heng Percival Zhang, Bamboo saccharification through cellulose solvent-based biomass pretreatment followed by enzymatic hydrolysis at ultra-low cellulase loadings, *Bioresour.*, **101(13)**, 4926-4929 **(2010)**

27. Sunarti T.C., Yanti S.D. and Ruriani E., Two-steps microwave-assisted treatment on acid hydrolysis of sago pith for bioethanol production, IOP Conf. Ser. Earth Environ. Sci., **65(1)**, 012052 **(2017)**

28. Thangavelu, Kannan Saravana, Rajkumar T., Pandi Dinesh Kumar, Ahmed Abu Saleh and Ani Farid Nasir, Microwave assisted acid hydrolysis for bioethanol fuel production from sago pith waste, *Waste Manag*., **86**, 80-86 **(2019)**

29. Van Soest, Pc J. and JcB Robertson, Proceedings, IDRC, Ottawa, ON, CA **(1979)**

30. Yemiş Oktay and Giuseppe Mazza, Acid-catalyzed conversion of xylose, xylan and straw into furfural by microwave-assisted reaction, *Bioresour*., **102(15)**, 7371-7378 **(2011)**.

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