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Conversion of hemicellulose from kenaf core fiber to xylose through dilute sulfuric acid hydrolysis

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Abstract

Kenaf (*Hibiscus cannabinus*) is a lignocellulosic plant that is usually utilized as a fiber source for sack production. The core from kenaf fiber has not been utilized yet in Indonesia, therefore it is still considered as a waste. Hemicellulose from kenaf core can be hydrolyzed to xylose through dilute sulfuric acid hydrolysis in high temperature. Hydrolysis in this study was done by using autoclave at 121℃ and 10% (m/v) biomass: acid ratio for 15 and 45 minutes with a variation on acid concentration (2%, 4%, and 6% v/v). Xylose concentration in the hydrolyzate tends to increase with higher acid concentration and longer heating time. 6% (v/v) sulfuric acid concentration and 45 minutes of heating time produce the highest xylose concentration (20.53 gr/L) and yield (86.50%).

1. INTRODUCTION

Utilization of renewable and eco-friendly resources is a necessity for creating a sustainable global environment. With a global production rate of approximately 200 billion tonnes per annum, lignocellulosic biomass is regarded as one of the most promising sustainable resources to be exploited [1]. Unlike other types of biomass such as sugar or starchy plants, utilization of lignocellulosic biomass will not create a disturbance on food supply [2]. Lignocellulosic biomass can be collected from agricultural wastes, such as corn stover, bagasse, and empty fruit bunch [3]. Aquatic plants such as seaweed and microalgae can also be used as a source of lignocellulosic biomass [4, 5]. Sophisticated technologies can convert lignocellulosic biomass into a high-value commodity such as biofuels, energy sources, and chemicals [6].

Kenaf (*Hibiscus cannabinus*) is a lignocellulosic plant that is grown for many applications, such as pulp materials, polypropylene composite, fiberboards, and insulations [7]. Kenaf currently has approximately 3,000 hectares of plantation area in Indonesia with a harvest time of 4 months [8]. Kenaf fiber is composed of 2 distinct parts, the hard outer bast and the soft inner core [9]. The core fiber of kenaf has not been utilized yet in Indonesia, therefore it is still considered as a waste. The yield of core fiber from kenaf plantation is approximately 6-8 ton/hectare, therefore Indonesia can produce about 54,000-96,000 tonnes of kenaf core fiber per year [10]. This agricultural waste can be exploited to increase the value of kenaf cultivation in Indonesia. Utilization kenaf core waste also serves as a waste valorization that helps in creating a sustainable, zero-waste community that converts agricultural waste into useful products [11].

Lignocellulose is composed of three primary bio-polymers: cellulose, hemicellulose, and lignin. Utilization of lignocellulosic biomass requires hydrolysis reaction to break cellulose and hemicellulose into useful products, such as glucose and xylose [12]. The hydrolysis reaction of lignocellulose can be performed with a diluted sulfuric acid solution under high temperature [3].

This method is favored because of its simplicity, cheapness, and high capability to hydrolyze hemicellulose its monomers directly without requiring other processes such as lignin degradation or enzymatic hydrolysis [13, 14]. Also, using dilute sulfuric acid reduce the formation of side products such as acetic acid and furfural [6].

Xylose ($C_5H_{10}O_5$) is a sugar compound that is commonly used as a sweetener in food and medicine [15]. Xylose can also be used as a precursor for producing numerous useful chemicals such as bioethanol, acetone, xylitol, and butanol [16, 17, 18]. Xylose is produced by hydrolyzing xylan, one of the hemicelluloses on plants [19]. While xylose can also be produced using enzymatic hydrolysis, acid hydrolysis is more competitive because it does not require any pre-treatment procedures and can produce xylose much faster than using enzyme [20].

Production of xylose through dilute sulfuric acid hydrolysis had been researched on many lignocellulosic biomasses, each yielding satisfactory result. On bamboo timber, hydrolysis with 2% sulfuric acid at 180°C for 40 minutes produced 0.7-1.2% xylose [21]. Xylose yield of 83.2% was produced from rice straw using 1% sulfuric acid at 100°C for 5 reflux cycle [22]. Hydrolysis of corn stover using 1% sulfuric acid at 140°C for 15 minutes produced 75.7% of xylose yield [23].

In this study, kenaf core fiber was used as a raw material to produce xylose through hydrolysis using three variations of sulfuric acid concentration and two variations of heating time. The cellulose and hemicellulose mass flow and loss through the hydrolysis process will also be evaluated. The effects of the variations to the yield and concentration of xylose will also be evaluated.

2. EXPERIMENTAL SECTION

2.1. Materials

Kenaf core fiber used was obtained from BALITTAS (Balai Penelitian Tanaman Pemanis dan Serat/Research Association of Sweetener and Fiber Plants), Malang, East Java, Indonesia. Kenaf core was sieved until the average particle size of approximately 6.5 mm and dried until the moisture content of approximately 8.60% as was measured by Moisture Analyzer OHAUS MB 45. Sulfuric acid from Merck EMSURE® (1.00731 EMD Millipore) was used in the hydrolysis. All other reagents and chemicals were analytical grade.

2.2. Procedure

2.2.1. Sulfuric Acid Hydrolysis

The hydrolysis was conducted with a biomass loading (ratio between biomass and solution) of 10% w/v (200 gr kenaf core + 2,000 mL sulfuric acid solution). The sulfuric acid concentrations are 2%, 4%, and 6% (v/v). Hydrolysis was conducted by heating in LabTech LAC-2031SN Autoclave at 121°C for 15 and 45 minutes. The hydrolyzate/filtrate was then separated from the solid residue by filtration with a hydraulic press filter. The residue was washed with water to pH 5-6 and dried in the oven at 50°C until the moisture content is below 10%. Analytical procedure for the measurement of the lignocellulosic component on the residue was provided by the National Renewable Energy Laboratory (NREL) [24]. The filtrate was neutralized to pH 7.0 by adding NaOH pellets, then has its xylose and glucose content analyzed with HPLC analysis.

The yield of xylose and glucose ($\%Y_s$) was calculated by comparing the mass of measured sugar in the hydrolyzate ($m_{s,m}$) with the theoretical mass of sugar from the biomass ($m_{s,t}$). The theoretical mass of sugar is calculated based on an assumption that all cellulose and hemicellulose in the biomass was hydrolyzed into glucose and xylose, respectively. The yield was calculated using the following equations:

$$\%Y_s = \frac{m_{s,m}}{m_{s,t}} \times 100\% \tag{1}$$

$$m_{s,t} = m_{c,u} \times \text{Anhydro correction} \tag{2}$$

$m_{c,u}$ is mass of cellulose or hemicellulose in the untreated sample. Anhydro correction is 1.11 for cellulose to glucose or 1.14 for hemicellulose to xylose [24].

Mass flow of the process was evaluated by calculating the mass of cellulose and hemicellulose before and after the hydrolysis process. The mass of cellulose and hemicellulose before the process can be determined using the result of component analysis on the untreated kenaf core fiber. On the other hand, the mass of cellulose and hemicellulose after the process can be determined by combining the result of HPLC analysis on the hydrolyzate and the result of component analysis on the solid residue of the hydrolysis process.

The mass of cellulose and hemicellulose before the process ($m_{c,b}$) can be calculated using the equation:

$$m_{c,b} = \%C_u \times \text{mass of substrate} \tag{3}$$

$\%C_u$ is the percentage of cellulose or hemicellulose of the untreated sample and mass of substrate is the mass of the untreated sample before the hydrolysis (dry basis).

The mass of cellulose and hemicellulose after the process ($m_{c,a}$) can be calculated using the equation according to Rocha, et al. [25].

$$\begin{aligned} m_{c,a} &= \text{Component mass in residue} \\ &\quad + \text{Component mass in filtrate} \\ &= (\%C_r \times \text{mass of residue}) \\ &\quad + (S_h \times V_h \times \text{Anhydro correction}) \end{aligned} \tag{4}$$

where $\%C_r$ is the percentage of cellulose or hemicellulose in the solid residue, the mass of residue is the mass of solid residue after the hydrolysis (dry basis), S_h is the concentration of glucose or xylose in the hydrolyzate, and V_h is the volume of the hydrolyzate.

The sample codes based on the used variations are shown in Table 1.

Table 1. Sample Codes

Sample Code	Sulfuric Acid Concentration (%v/v)	Heating Time (minutes)
A	2	15
B	4	15
C	6	15
D	2	45
E	4	45
F	6	45

2.2.2. Products Analysis

Glucose and xylose products were measured by High-Performance Liquid Chromatography (HPLC) Waters, USA. The mobile phase is 5 mM H_2SO_4 at 0.6 mL/min and the stationary phase is Bio-Rad Aminex HPX-87H column. The detector is a Waters 2414 refractive index (RI) detector. The oven temperature was maintained at 40°C in the column input and 65°C in the output.

3. RESULT AND DISCUSSION

Kenaf used in this study contained 37.77% cellulose, 13.66% hemicellulose, and 32.31% lignin. This was compared with the chemical component reported by Tye et al. [9] (52.8% cellulose, 47.2% hemicellulose, and 21.4% lignin), the raw material had lower cellulose & hemicellulose content and higher lignin content. There are many factors that could affect the lignocellulosic content of a single plant species such as age, soil types, climate, and nutrition [26].

3.1. Hydrolyzed Lignocellulosic Content

The lignocellulosic content of the raw and hydrolyzed samples of kenaf core fiber is shown in Table 2.

Table 2. Lignocellulosic content of the raw and hydrolyzed samples of the kenaf core fiber

Sample Code	Cellulose (%)	Hemicellulose (%)	Lignin (%)
A	47.80	4.18	35.78
B	50.10	3.04	36.48
C	49.30	0.44	37.98
D	50.75	2.34	35.61
E	49.54	0.29	39.54
F	48.97	0.09	39.31

Higher acid concentration or longer heating (both of these factors are defined as ‘reaction severity') tends to produce lower hemicellulose content in the hydrolyzed solid residue. A similar tendency was obtained by Rocha et al. where hemicellulose content in hydrolyzed CAB (cashew apple bagasse) tends to decrease with higher acid concentration, ranging from 5.3% with 0.2 M sulfuric acid to 2.6% with 0.8 M sulfuric acid [25]. This tendency is expected since sulfuric acid is targeted to hydrolyze the hemicellulose in lignocellulosic material into xylose. Lower hemicellulose residue means that the hydrolysis occurred to a greater extent, which is something to be expected with the usage of higher acid concentration and longer heating time.

Contrary to what happened to the hemicellulose content, cellulose and lignin fraction tends to increase after the hydrolysis. A similar tendency was obtained by Li et al., where hydrolysis on bamboo timber with A sulfuric acid concentration of 2% and biomass loading of 1:6.25 w/v at 180°C decreased the hemicellulose content from 22.4% to 0.5% [21]. On the contrary, the cellulose and lignin content in that study was increased from 46.5% to 63.9% and from 23.3% to 37.4% [21]. The increased cellulose and lignin content indicated that sulfuric acid tends to exclusively hydrolyze hemicellulose into xylose with little to negligible effect on cellulose and lignin.

The lowest hemicellulose residue in this study (0.09%) is notably lower compared to the obtained results in other studies. Rocha et al. only managed to reduce the hemicellulose content of cashew apple bagasse from 16.3% to 2.6% with the usage of 0.8 M sulfuric acid at 121°C for 30 minutes with a biomass loading of 15% w/v [25]. Li et al. reduced the xylan content of bamboo timber from 21-23% to 0.4-0.7% using 2% sulfuric acid at 180°C for 40 minutes [21]. The high hemicellulose removal could be attributed to the brittleness and discreteness of the kenaf core fiber particles. Brittleness allows easier acid penetration into the hemicelluloses inside the biomass while the discreteness allows the higher area of contact between the biomass and the acid solution, therefore enabling more xylose to be produced.

Effective hydrolysis of glucose into cellulose usually requires special cellulase enzyme or very high reaction severity while lignin degradation usually requires other treatment methods such as alkaline treatment or steam explosion [27, 28]. Unfortunately, physical methods are usually incompatible with sulfuric acid due to its high corrosiveness. The usage of physical methods which usually utilize high temperature and pressure can also trigger xylose degradation and unwanted side reactions that produce toxic chemicals, such as formic acid, acetic acid, and furfural [29]. One of the solutions to solve this incompatibility is by performing acid hydrolysis on lignocellulosic biomass to produce xylose, followed by enzymatic hydrolysis on the solid residue to produce glucose. This method was done in a study by Camesasca et al. on corn stover as the raw material and managed to produce 95.54% as the highest xylose yield and the highest glucose concentration of 2.7% [30].

3.2. Xylose Concentration & Sugar Yield

The profile of xylose concentrations across different sulfuric acid concentrations and heating time are shown on Fig.1. and Fig.2.

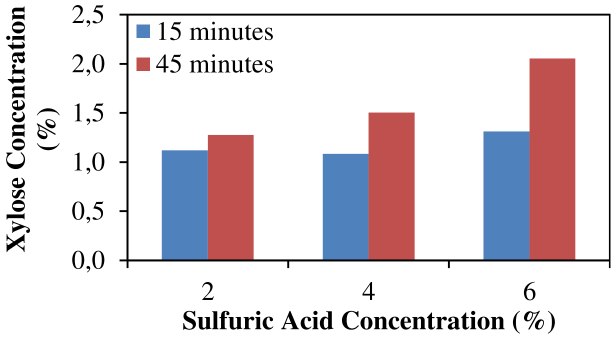

Fig.1. Xylose concentrations for different sulfuric acid concentrations

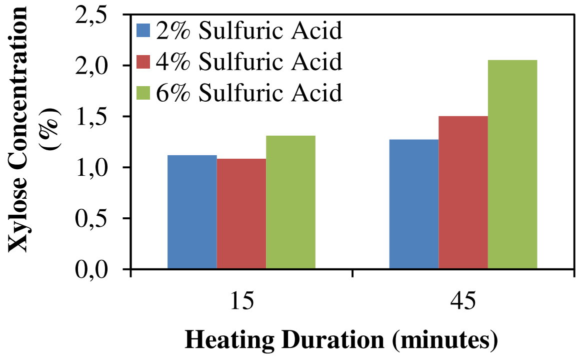

Fig.2. Xylose concentrations for a different heating time

Both of these figures show that the increase of xylose concentration tends to rise along with increasing sulfuric acid concentration and longer heating duration. A similar tendency was acquired by Kumar et al. where the increase of sulfuric acid concentration from 2% to 4% increased the produced xylose concentration from 0.9 to 1.3%, while the usage of 6% sulfuric acid managed to increase the xylose concentration from 1.3 to 2.0% [31]. This tendency implies that the increase of acid concentration and heating duration synergically increased the produced xylose from the hydrolysis.

The sugar yield and from the hydrolyzate samples is shown in Table 4.

Table 4. Sugar (xylose and glucose) yield from hydrolyzate

Sample Code	Yield (%)	
	Xylose	Glucose
A	44.12	0.84
B	43.69	0.00
Sample Code	Yield (%)	
	Xylose	Glucose
C	62.17	1.62
D	49.74	2.52
E	73.53	3.73
F	86.50	4.68

Higher reaction severity tends to produce higher sugar yield. A similar result was obtained by Rafiqul et al. where xylose recovery obtained from acid hydrolysis on *Meranti* wood sawdust tend to increase with higher reaction severity, reaching a maximum value of 92.05% with the usage of 4% sulfuric acid on 125°C for 60 minutes [32]. During dilute acid hydrolysis, the acid molecule diffuses from the bulk solution to the pore space in the biomass. Once the acid molecule reached the reactive sites, it acts as a proton source that will initiate the breakage of glycosidic linkage of the cellulose or hemicellulose polymers to form monomeric sugars [27]. Higher acid concentration means more acid molecules that could act as a proton source while longer heating duration means longer time for the hydrolysis reaction to proceed, therefore increasing the extent of the hydrolysis and the production of monomeric sugars

Hydrolysis performed in this study produces much more xylose than glucose. This result is comparable to Hong et al. where xylose production rate using dilute sulfuric acid on corn stover can reach up to 82.8% on the optimum condition, while glucose production rate can only reach 26% [33]. This tendency happened due to the molecular structure difference between cellulose and hemicellulose. Cellulose polymers tend to be straight and long-chained while hemicelluloses tend to be amorphous and short-chained, making hemicellulose easier to be broken down into its monomer than cellulose [34].

The highest xylose yield and concentration obtained in this study are 86.50% and 2.05%, respectively. Both of these results were produced from the sample with the highest reaction severity (meaning the highest sulfuric acid concentration and longest heating duration). Compared to the results of other studies, the xylose yield obtained from this study is quite satisfactory considering the biomass loading is quite low (10% w/v). This result is similar with the obtained result from Kumar et al.; where the usage of 6% sulfuric acid and biomass loading of 12.5% w/v produced 2.0% xylose from sugarcane bagasse [31]. However, the obtained xylose yields from that study were quite low, ranging from 13-38% [31]. Rocha et al. produced 3.6% of xylose from cashew apple bagasse with the usage of 0.6 M sulfuric acid and biomass loading of 30% w/v [25]. However, this optimum result only produced xylose yield of 83% [25].

3.3. Cellulose and Hemicellulose Component Losses

Component losses in this study were defined as the discrepancy between the mass of cellulose and hemicellulose before the process ($m_{c,b}$) and after the process ($m_{c,\,a}$). The percentage of cellulose & hemicellulose component loss through the process from all the samples is shown in Table 3.

Table 3. Cellulose and hemicellulose component losses during hydrolysis

Sample Code	Component Loss (%)	
	Cellulose	Hemicellulose
A	12.26	34.84
B	21.22	43.10
C	26.52	36.05
D	20.92	40.51
E	30.09	25.40
F	29.18	13.15

It is shown that cellulose component loss tends to increase with the usage of higher acid concentration and longer heating duration. Component losses can be caused by partial hydrolysis that produces soluble oligomers, sugar degradation that produces HMF and furfural, or side reactions that produce organic acids [27]. These compounds are undetectable by HPLC analysis or lignocellulosic component analysis, meaning that its amount cannot be measured and its formation will contribute to the component losses. Higher reaction severity will also increase the extent of the side reactions, meaning that more unwanted products will be produced.

Hemicellulose component losses exhibit different tendency compared to cellulose. The usage of 4% and 6% sulfuric acid with 45 minutes heating duration produced hemicellulose component losses to 25.40% and 13.15%; while hemicelluloses component losses from other samples range between 34-43%. Hemicellulose hydrolysis begins with partial hydrolysis that converts hemicellulose polymers into soluble oligomers, followed by complete hydrolysis that breaks down soluble oligomers into xylose monomers [27]. This different tendency indicates that higher reaction severity promotes complete hydrolysis of hemicellulose to xylose, leading to more soluble oligomers being converted into xylose, causing a decreasing amount of oligomers and increasing amount of xylose that leads to lower calculated component losses.

3.4. Cellulose and Hemicellulose Mass Flow Analysis

The calculation result of cellulose and hemicellulose mass flow during sulfuric acid hydrolysis of sample F is shown on Fig. 3. It can be seen that the total cellulose and hemicellulose in the output is less than in the untreated sample/input. The possibilities of component losses are highlighted in Fig. 3. Hydrolyzate loss can occur during filtration since the produced residue will always contain some of the hydrolyzates. Considering that the washing process uses a very large amount of water compared to the volume of the residue, this hydrolyzate can be washed off along with a small portion of the residue during the washing process. Another residue loss also occurs after the washing process, specifically when the washed residue is moved from the filter cloth to the oven tray.

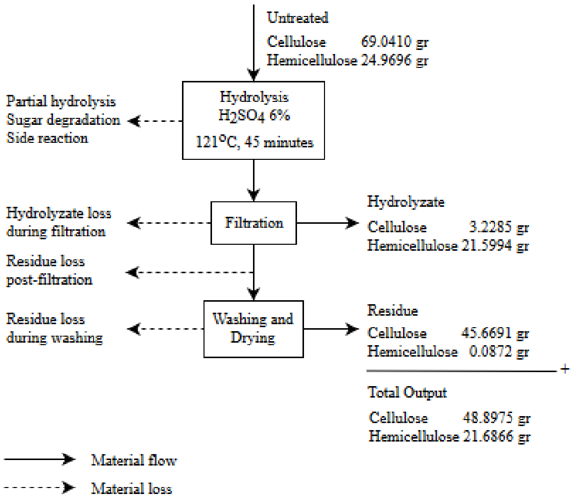

Fig 3. Cellulose and hemicellulose mass flow of during sulfuric acid hydrolysis of sample F

The solid residue from the hydrolysis contains very little hemicellulose (0.0872 gr) compared to cellulose (45.6691 gr). The hydrolysis removed almost all the hemicellulose in the kenaf core fiber, leaving only 0.35% of the untreated hemicellulose content as the residue. This low residue indicates that the performed condition (6% sulfuric acid in 121°C for 45 minutes) is already quite effective at converting almost all of the hemicellulose in the kenaf core fiber. Conversely, hydrolyzate contains much more hemicellulose (in form of xylose) and cellulose (in form of glucose). Xylose is a highly-soluble compound, meaning that it will be instantly dissolved into the hydrolyzate once it was formed during the hydrolysis. On the other hand, cellulose is a recalcitrant and relatively insoluble polymer that will not be dissolved into the solution unless it is hydrolyzed into glucose [28].

4. CONCLUSION

Hydrolysis of kenaf core was carried out with dilute sulfuric acid to obtain xylose in the hydrolysate. It was found that under 6% sulfuric acid for 45 min conditions could produce 20.53 gr/L xylose and 86.50% yield. The hydrolysis process where the best sample that produced the highest xylose yield also produced the lowest hemicellulose loss, meaning that it represented the most efficient hydrolysis condition in this study.

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