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Sensitivity analysis of xylose production process using aspen plus

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Abstract. Xylose production has become one of the most studied process over the year due to the significant application as a raw material for the production of a variety of specialty chemicals, mainly xylitol. The most promising raw material is lignocellulosic biomass because of its widely available and cheap. There are several type of pretreatment process has been studied to depolymerized the lignocellulosic compounds into fermentable sugars. Among all, dilute acid hydrolysis is the most promising process to produce high xylose. However, disadvantage of this pretreatment process is production of byproduct that can slowdown the fermentation step. Understanding the effect of pretreatment processing parameters on lignocellulosic depolymerization could possibly result in minimization of degradation compounds. Therefore, the aim of this work is to carry out the sensitivity analysis of the acid hydrolysis process used for the production of fermentable sugars with the aid of Aspen Plus by considering the concentration of xylose, glucose, furfural and acetic acid obtained at the outlet of reactor as the output variable. Sensitivity analysis were apply at temperature (160 °C, 170 °C and 180 °C) and residence time (0-160 min). The results indicate that the developed process model is possible to improve lignocellulosic conversion efficiency while minimizing degradation product generation with the highest xylose produce is 18.26 g/L at 180 °C during 20 min reaction time.

1. Introduction

Lignocellulosic materials and hydrolysis process are very complex, it was observe that release of xylose, glucose, and furfural in the hydrolysate was dependent on experimental operating conditions. The acidity of the system, type of lignocellulosic material, solid-to-liquid ratio, operating temperature and time, rate of decomposition of hydrolysis products and the type of reactors are seems to be major influence on hydrolysate concentration. Among those parameters, temperature, reaction time, acid concentration and type of lignocellulosic materials are the most widely investigated factors. [1] Prove that the efficient of hydrochloric and sulphuric acid concentrations for hemicellulose hydrolysis are in the range of 0.5-1.5 % and temperatures between 121 °C and 160 °C. However, there is no direct principle or method, which were consider the same for all lignocellulosic materials. [2]In his study found that, the effect of temperature is more important compared to acid concentration and time while [3] found that the acid concentration is the most significant factor for hemicellulose hydrolysis. In general, dilute acid hydrolysis using mild process conditions cause significant sugar degradation and thus yield high hydrolytic efficiency and increase the amount of fermentable sugars recovery. The main problem in this

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study is to study the effect of temperature, time and acid concentration on xylose recovery using kinetic model. Although studies have been develop to analyse the flexibility of hydrolysis process with respect to temperature, time and acid concentration in xylose recovery, those studies were perform under different operating conditions and simulation technique. That is, the kinetic aspects of hydrolysis reaction was not included in those studies. Therefore, the ability of sensitivity analysis using Aspen plus to operate within limits is feasible to overcome the problem.

Process simulation is becoming an important tool in both bioprocess and petrochemical industries. Simulation tools were use at any stage of process development, from initial concept, through design, to final plant operation. These tools tackle a range of tasks, including creating process flow diagrams, generating material and energy balances, determining equipment sizing, and estimating capital and operating costs. Aspen Plus simulator is one of powerful computation tool for pretreatment study. Research on simulation of lignocellulosic pretreatment process using Aspen Plus simulator was extensively study in the last two decades [4, 5, 6, 7 and 8]. In Aspen Plus simulator, it contains the function of sensitivity analysis in the model analysis tools in order to analyse the simulation process. This tool used to determine how a process reacts by varying the key operating and design variables [9]. Sensitivity analysis has several benefits among which are studying the effects of changes in input variables on process outputs, verifying that a solution to a design specification is feasible, graphically representing the effects of input variables, and studying time varying variables using a quasi-steady state approach [10]. The advantages of this tool offer the opportunity to study the effect of process parameter of pretreatment dilute acid hydrolysis process interactively in a short time. The response of key process variables such as concentration of xylose, glucose and furfural due to changes in temperature and time were analysed and presented in this study. Generally, higher temperature is preferable for cellulose hydrolysis and longer hydrolysis process will result in decreasing xylose concentration and increasing glucose and furfural concentration.

2. Kinetics of acid hydrolysis process

The hydrolysis reactions using dilute acid are very complex, mainly because the presence of strong bonds in the raw material, the different phase of substrate (solid) and catalyst (liquid) and strong protection against the attacks of chemical to the structure of the whole cells [11, 12]. A number of mathematical model have been report in the literature to represent the kinetic of acid hydrolysis process of a variety of lignocellulosic biomass [11, 12, 13 and 14]. Most of the kinetic models based on an irreversible pseudohomogeneous first order reaction. These models were first propose by Saeman for the hydrolysis of wood using sulphuric acid [15]. The Saeman's model was also apply to the hydrolysis of the hemicellulose fraction, which is represented as

Xylan $\xrightarrow{k_1}$ Xylose $\xrightarrow{k_2}$ decomposition products

where k1 and k2 is the rate of xylose formation and the rate of decomposition of xylose respectively. From this reaction model and solving differential equations, xylose concentration as a function of time represented by:

$$[Xc] = \frac{(k1Xnp)(e^{-k_1t} - e^{-k_2t})}{k_2 - k_1} + (Xo)e^{-k_2t}$$
(1)

where Xc is xylose concentration (g/L), Xnp is xylan concentration (g/L) and Xo is initial xylose concentration (g/L).

Assuming that initial xylose concentration to be approximately equal to zero, then equation (1) can be simplify to:

$$[Xc] = \frac{(k_1 Y n p) (e^{-k_1 t} - e^{-k_2 t})}{k_2 - k_1}$$
(2)

The kinetic model of cellulose hydrolysis to monomer glucose also involves a series of irreversible pseudohomogeneous first order reaction and represented as:

Cellulose
$$\stackrel{k_3}{\rightarrow}$$
 Glucose $\stackrel{k_4}{\rightarrow}$ decomposition products

where k3 and k4 is the rate of conversion of cellulose to glucose and the rate of decomposition of cellulose respectively. Due to unfavourable operating conditions for glucose degradation, the decomposition reactions were negligible in this study. Therefore, the model can be simplify to:

Cellulose
$$\stackrel{k_3}{\rightarrow}$$
 Glucose

From this reaction model and solving differential equations, glucose concentration as a function of time can be represent by:

$$[Gc] = [Gp](1 - e^{-k3t})$$
(3)

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where Gc is Glucose concentration (g/L), and Gp is Cellulose concentration (g/L).

In the hydrolysis of lignocellulosic materials, furfural (decomposition products of pentoses) and phenolic compounds (decomposition product of lignin) are generate. Generally dilute acid hydrolysis process affects hemicelluloses and only strict conditions will affects cellulose [16]. Therefore, the amount of phenolic compounds in hydrolysate is negligible and the experimental results reported by [17] is consider as furfural. Hence, the kinetic model for furfural concentration [Fc] as a function of time can be express as:

$$[Fc] = [Fp](1 - e^{-k5t})$$
(4)

where Fc is Furfural concentration (g/L), Fp is Potential concentration of furfural (g/L) and k5 is Furfural formation rate (min⁻¹).

3. Materials and method

The Aspen Plus model used for the accomplishment of the xylose process shown in figure 1. The model is based on the several assumptions: steady state operation; the biomass input stream consist of pure elemental solid which are xylan, cellulose and lignin; fast hydrolysis was consider and only xylose and glucose was produced from the xylan and cellulose breakdown. The steady state simulation employing the following stages: mixing, heating, dilute acid hydrolysis reactor, cooling, neutralization reactor and solid separation was apply to validate the simulation results with the experimental study by [16]. NRTL (Non-Random Two liquid) model was select as a property method owing to system characteristic which is operation pressure lower than 10 atm and system compounds are no-electrolyte polar substance [12]. The steady state simulation of xylose production is required to identify a reasonable range of decision variables before sensitivity done. For steady state simulation, two inlet stream; sawdust and dilute sulfuric acid (6% w/w) were mixed and the temperature were heat up to 105 °C, 115 °C , 125 °C and 130 °C in a heat exchanger (heater). The outlet solution are then inlet to kinetic reactor (R-01) for 60 minutes hydrolysis process. The hydrolysate solution of the reactor were cool down to 25 °C using heat exchanger (cooler) prior to filtration in order to facilitate gypsum formation and removal from hydrolysate solution. The hydrolysate solution are then feed to conversion reactor for neutralization process where calcium oxide flakes were added at 25 °C and atmospheric pressure and the acid-base reactions took place to form salt and water. The input of conversion value were calculated based on initial content of xylan (29g xylan/100g MWS on o.d.b.) and cellulose (41 g cellulose/100g MWS on o.d.b) in MWS. The remaining sulfate in the hydrolysate solution were then separate using solid

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separator (separator) prior to fermentation process. The complete process model was validate at steady state using experimental data providing by [16] while initial guesses for split fraction value for liquid solid separator unit were determined from the process design for the biomass-to-ethanol process utilizing co-current dilute acid prehydrolysis with simultaneous saccharification and co-fermentation report by NREL.

A number of other important kinetic information can be derived by successfully determined the accurate k values for the reactions through the mathematical models. By applying the Arrhenius Equation, the kinetic coefficients and activation energy were obtain. This correlation is indicating below;

$$k_{\rm i} = k_{\rm i0} e^{\frac{-Ea}{RT}} \tag{5}$$

where;

 k_i = Kinetic coefficient (min⁻1) k_{i0} = Pre-exponential factor (min⁻1) Ea = Activation energy (kJmol⁻1) R = Gas constant, 8.314 (kJmol⁻1) T = Temperature (K)

From the above equation, the reaction constant k_i increases exponentially with temperature. In order to calculated the activation energy, Ea, equation (5) must be rearrange into a linear function form; y = mx + c, whereby the reaction coefficients are represented by the linear constants *m* and *c*. Equation (5) are therefore rearrange as below;

$$\ln k_{\rm i} = -\frac{Ea}{RT} + \ln k_{\rm i0} \tag{6}$$

Having successfully calculated all the reaction constant, k_i , it is now possible to determine the activation energy at different temperature. Table 1 shows the reaction constants from [18] and activation energy obtained from equation (6) for all sugars investigated at 0.2% H₂SO₄ concentrations.



Figure 1. Flowsheet of model development process for the Xylose production using Aspen Plus.

Sensitivity analysis of hydrolysis process was do once minimum error was achieve in model validation. As the dilute acid hydrolysis unit stated to be the core component in this study, a section of sensitivity analysis was, apply for this process unit. The development of the model was carry out by selecting the Continuous Stir Tank Reactor (CSTR) owing to the kinetic reaction that based on the rate constant and activation energy. Methods used during sensitivity analysis were describe with emphasis on sampled flow sheet variables, varies flow sheet variables, varied variables and tabulated variables. In

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Aspen plus, sensitivity analysis was performed to determine how a process reacts to varying key operating and design variables. In order to optimize the dilute acid hydrolysis process, sensitivity analyses were set to study the hydrolysis performance. Simulation trials were conduct by varying one operating parameter while the others remained the same. In this simulation, the reactor temperature was varied from 160 $^{\circ}$ C, 170 $^{\circ}$ C and 180 $^{\circ}$ C and residence time was varied from 0 -160 min while acid concentration was remained constant (0.2%).

4. Result and discussion

The simulation was develop on the basis input data from a standard experimental run of the xylitol production by [16]. The experimental input data for the feed streams, kinetic reaction, conversion rate and operating conditions used as input data for the simulation. The simulation results were validate for all fermentable sugars product through comparison with literature [16]. The purpose of this validation step is to find the efficiency of the process model and to determine whether the applied experimental parameters was able to simulate the dilute acid hydrolysis process. Validation was done by calculate the overall mean error of the simulation result with the experimental result. The process model validation for the complete process model in term of product concentration in the stream outlet (S-5) have proven to be in reasonable agreement with experimental study. The sensitivity analysis of Continuous Stir Tank Reactor model with the diluted acid hydrolysis process used for the production of fermentable sugars; xylose, glucose and furfural shown in figure 3 and 4, respectively for the concentration profiles.

	160 °C	170 °C	180 °C
Xylose			
$k_1 (\min^{-1})$	0.0209	0.0340	0.0863
E_1 (kJ/mol)		115.41	
Glucose			
$k_3 (\min^{-1})$	0.0057	0.0070	0.0108
E_3 (kJ/mol)		51.99	
Furfural			
$k_5 (\min^{-1})$	0.0196	0.0219	0.0266
E_5 (kJ/mol)		24.86	

Table 1. Reaction constant and activation energy of sugars released at different temperature (160 - 180° C) and constant H₂SO₄ concentrations (0.2%).

4.1. Xylose production

Xylose is the main product generate when choosing dilute acid for hydrolysis process. To study the effect of temperature and residence time on the production of xylose, three temperature levels (160 °C, 170 °C and 180 °C) were selected. Fermentable sugar product through concentration of xylose at different times and temperature shown in figure 1 in S-5 streamline. The significant of this method is to study the relation of kinetic parameter (reaction constant and activation energy) with the temperature and time towards the products concentration. The reaction time was varies from zero to 160 minute. A representative plot in figure 3 shown that the concentration of xylose at different time and temperature behaves differently when compared with other products. It was observe that the concentration of xylose increased with increasing temperature, reached to a maximum value of 18.26 g/L at 180 °C for 20 min residence time, and then experience a progressive decrease. This was due to decomposition reactions and production of inhibitor that occurs as hydrolysis continues. The lowest xylose produced was 6.67 g/L at 160 °C. 16.45 g/L and 8.43 g/L of xylose was release at 180 °C for 40 and 140 min residence time. The same trend is observe for temperature of 170 residence time. At these temperatures, approximately 30% of xylan conversion occurred within the entire reaction. The corresponding of xylose concentration arrived at a higher value of 13.76 g/L and 11.65 g/L after 40 min reaction time for respective temperature. Results indicated that longer residence time and high temperature is not essential

for higher xylose recovery. A relatively shorter residence time around 60 min is favourable for the maximum xylose recovery and minimum by products degradation. Results indicated that longer residence time and high temperature is not essential for higher xylose formation as severe hydrolysis condition may lead to formation of by-products. These proved that reaction temperature is a key parameter effects on the conversion of xylan to xylose and higher temperature could shorten the time required for highest xylose formation, which was consistent with Choudhary et al. findings.



Figure 2. Concentration of Xylose at different residence time and various temperature at 0.2% H₂SO₄ concentrations.

4.2. Glucose production

Glucose is a main by product released in acid hydrolysis of sawdust. Glucose can be released from both components; hemicellulose and cellulose. Glucose are mostly release from cellulose under strict operational conditions and only small amount of glucose was hydrolyse in the range of operational conditions commonly used for the dilute acid hydrolysis. However, in this analysis the released of glucose are quantitatively from cellulose due to the reaction implement in simulation. Therefore, the hemicelluloses (xylan) conversion are not considered. Glucose concentration were constantly increase as the reaction time and temperature increase. A representative plot is display in figure 4. The highest glucose released is 24.30 g/L at 180 °C for 160 min reaction time. At this operational condition, 67% of cellulose to glucose occurred with approximately 22% conversion. As residence time increase (40 -160 min) the conversion is greatly increased with approximately 34%. The obtained glucose throughout the process is mainly comes from cellulose, which is susceptible to the hydrolysis. The degradation of cellulose will begin to contribute to the generation of glucose as the reaction continues and high temperature is applied.



Figure 3. Concentration of Glucose at different residence time and various temperature at 0.2% H₂SO₄ concentrations.

4.3. Furfural production

A representative plot for furfural concentration displayed in figure 4. The production of furfural was highly influence by the reactor temperature and residence time. At low temperature and shorter residence time, the furfural production is relatively low. However, the production is drastically increase with the higher temperature and longer residence time. Overall, the conversion of xylose to furfural is relatively low throughout the reaction time, which is less than 20%. The highest furfural concentration is 17.34 g/L obtained at 180 °C for 160 min residence time. This production will continuously increase, as furfural is the side product from xylose decomposition. The condition that minimizes the generation of furfural is favourable since furfural is an inhibitor of the growth of microbes for fermentation.



Figure 4. Concentration of Furfural at different residence time and various temperature at 0.2% H₂SO₄ concentrations

In general, higher temperature and acid concentration is preferable for cellulose hydrolysis and longer hydrolysis process will result in decreasing xylose concentration due to degradation to inhibitor [19]. Under this condition, xylan decomposed very fast to xylose and degraded completely to furfural. Obviously, from the figure 3 and 4 the temperature of 180°C and 0.2% acid concentration provides more preserving conditions for xylose, glucose and furfural in reaction solution. Limited xylose degradation observed. At the same time, the xylose formation (as a main objective) was high under this process temperature and time, pointing to complete xylan conversion. However, the concentration of glucose and furfural were low during 60 min of reaction time while xylose achieved the highest concentration within 20 min of reaction. This trend was similar to other studies by [20] where maximum xlose yields obtained under higher temperature of 140 °C to 160 °C within short reaction time (10 - 30 min). This can be related with the reaction constant used in this study where the reaction constant of xylose formation and furfural formation obeyed the following order; $k_1 > k_2$ over the temperatures (160 – 180 °C). The relative rate of xylose formation, with respect to furfural formation (k_1/k_2) increase with the increasing temperature. Thus, high temperature will favor the formation of xylose [21]. This conclusion proved the trend in product concentration observed in this study as well as the published information. The Ea value for xylose formation (115.41 kJ/mol) found to be significantly higher than furfural formation (24.86 kJ/mol) and glucose formation (51.99 kJ/mol). This indicated that the formation of xylose occurred more rapidly than the formation of glucose and furfural. It was evident in current study where high xylose concentration (18.26 g/L) was obtain at temperature of 180 °C within 20 min reaction time compared to glucose (7 g/L) and furfural (5.12 g/L). In addition, reported by [22], for the feedstocks with large ΔEa (Ea value between the reactions of xylose formation and decomposition), the small increase in temperature would be expected to significantly increase the yield of furfural. The larger difference of Ea value between the reaction of xylose and furfural formation found in this study significantly increase the concentration of furfural even for a small increase of temperature. In this

regard, considering the value of k_1 and Ea for xylose and furfural, relative high reaction temperature and reduced reaction time could be beneficial for the xylose formation.

5. Conclusion

The kinetic study proved that woody sawdust is a potential feedstock for high xylose production with xylose formation activation energy of 115.41 kJ/mol. Sensitivity analysis of wood sawdust was simulate at various operating temperatures (160 - 180 °C) under various reaction times (0-160 min) and constant acid concentration (0.2%). The optimum condition using this model are 180 °C for 20 min with acid concentration of 0.2%. Under these conditions, the obtained results are the following: xylose 18.26 g/L glucose 7 g/L and furfural 5.18 g/L.

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