



Investigation of conditions for dilute acid pretreatment for improving xylose solubilization and glucose production by supercritical water hydrolysis from *Quercus mongolica*



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ABSTRACT

The objective of this study was to prepare a feedstock, which xylose should be highly solubilized and most of the glucose remained in the solid fraction, for supercritical water hydrolysis (SCWH). Response surface methodology (RSM) with two independent variables (reaction time and sulfuric acid concentration) was adopted to find suitable conditions for pretreatment using a simple apparatus an autoclave for massive production. Based on the results of the RSM experiments, higher xylose contents were obtained in the liquid fraction after dilute acid pretreatment at 121 °C for 60 min with 6.82% H₂SO₄ (condition #8 (18.0%)) and at 121 °C for 102.3 min with 4% H₂SO₄ (condition #6 (16.0%)). To preventing corrosion of the apparatus, condition #6 was modified by prolonging the reaction time to 105 min, which led to a xylose content of 17.6% in the liquid fraction. Furthermore, structural degradation of the solid fraction was observed, indicating a positive effect for sugar production via SCWH.

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1. Introduction

The demand for fossil fuels has increased considerably owing to rapid industrialization. However, because of fossil fuel depletion and environmental problems (e. g. climate change), the development of sustainable and renewable resources that can reduce the extensive use of fossil fuels is required [1]. Lignocellulosic biomass has attracted interest as an alternative to fossil fuels because of its abundance and carbon-neutrality [2]. Cellulose, which is one of the major components of lignocellulosic biomass, has been considered as versatile material, and its hydrolyzed form, glucose, is used as a chemical precursor [3]. However, the cellulose fraction is covered by hemicellulose and lignin which act as a physical barrier for cellulase and prevent suitable glucose production [4]. To use cellulose in lignocellulosic biomass efficiently, pretreatment is

essential because it breaks down cellulose-hemicellulose-lignin linkages and increased the pore size to enhance enzyme accessibility [5].

Recently, supercritical water hydrolysis (SCWH) has emerged as a novel approach for glucose production owing to short reaction times without enzyme utilization [6]. However, the major drawbacks of SCWH are low selectivity for sugar degradation and significant oligosaccharide production [7]. Therefore, an additional hemicellulose fraction elimination process is required prior to SCWH to improving selectivity for glucose [8].

Dilute acid pretreatment, which commonly involved the use of sulfuric acid owing to its low price and good reactivity with lignocellulosic biomass, has been researched for several decades and is recognized as a powerful hemicellulose removal method [9]. Glycosidic bonds between hemicellulose and cellulose or hemicellulose itself are readily cleaved by hydronium ions, which originate from the acidic catalyst, during dilute acid pretreatment [10]. However, severe pretreatment conditions can cause further degradation of cellulose and force it into the liquid fraction, which can reduce the glucose yield of SCWH [11]. Therefore, suitable

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condition that can prevent excessive isolation of the cellulose fraction and lead to high elimination of the hemicellulose fraction should be investigated. In this respect, response surface methodology (RSM) with central composite design has been considered as a proper tool for determining suitable condition to achieve the above purpose.

In this study, dilute acid pretreatment was performed on *Quercus mongolica* which is a common source of lignocellulosic biomass in East Asia. An autoclave was selected for the reaction instead of a conventional stainless steel reactor because of its relatively large capacity and good temperature maintenance capabilities in mild conditions. RSM was adopted to search for suitable reaction condition for xylose solubilization. And, large quantities of the solid fraction were produced under a modified condition, which was reflected the experimental RSM results.

2. Materials and methods

2.1. Materials

The feedstock in this study was from *Quercus mongolica* and was generously supplied by the National Institute of Forest Science (Seoul, Republic of Korea). The feedstock was ground and sieved to obtain particles smaller than 0.5 mm. The *Q. mongolica* powder was stored in plastic bags at 4 °C, and the moisture content was less than 10%. The raw material consisted of 45.8% glucan, 18.6% xylan, 0.6% arabinan, 1.4% galactan, 1.8% mannan, 20.9% acid-insoluble lignin, 3.1% acid-soluble lignin, 2.1% extractives, and 0.1% ash.

2.2. Dilute acid pretreatment

Dilute acid pretreatment was performed using an autoclave (MLS-3020, Sanyo, Japan) with a 48 L capacity. In Erlenmeyer flasks, 50 g of milled *Q. mongolica* was mixed with 350 mL of an aqueous sulfuric acid solution. The reaction temperature was fixed at 121 °C, which was the maximum temperature of the autoclave. The reaction time and sulfuric acid concentration were between 17.7 and 102.3 min and 1.18% and 6.82% (w/w), respectively, based on the 2² factorial central composition design, as shown in Table 1. The internal temperature of the autoclave was increased over approximately 30 min until the target temperature was achieved. After the desired reaction time for the dilute acid pretreatment had elapsed, the flask was chilled using a quenching vessel. The solid and liquid

fractions of the pretreated slurry were separated using filter paper (No. 52, Hyundai Micro Co., Seoul, Republic of Korea). The liquid fraction was filtered using a 0.45 µm membrane filter (Advantec Co., Japan), while the solid fraction was not washed and was kept at 4 °C for SCWH.

2.3. Response surface methodology (RSM)

Response surface methodology based on 2² factorial central composition design (CCD) was performed using Design Expert 7.0.0 software (Stat-Ease, Inc., USA). Table 1 presents ten sets of reaction conditions composed with four axial points, and the central point (reaction time: 60 min; sulfuric acid concentration: 4%) was duplicated. Reaction time (X₁, min) and sulfuric acid concentration (X₂, %) were designated as reaction factors, while xylose content (Y₁, %) in the liquid fraction after dilute acid pretreatment was set as a dependent variable. The coded level of the CCD from each run corresponded to real independent variables as follows: (variable = value of central point/variation of coded level per one point): reaction time (min) = 60/30; sulfuric acid concentration (% w/w) = 4/2.

2.4. Chemical composition of the solid and liquid fractions

The acid-insoluble lignin (AIL) and acid-soluble lignin (ASL) contents in the raw material and solid fractions after the dilute acid pretreatment were determined using the Laboratory Analytical Procedure of the National Renewable Energy Laboratory (NREL) [12]. The water-insoluble solid (WIS) recovery rate of solid fraction was measured by oven-drying at 105 °C for 24 h. The ash content in the raw material was estimated according to the Determination of Ash in Biomass [13].

The contents of sugars (glucose, xylose, arabinose, galactose, and mannose) in the raw material, solid fraction, and liquid fraction after the dilute acid pretreatment were determined using ion chromatography (ICS-5000, Dionex, USA) using the filtrate obtained after the AIL and ASL content measurement. The ion chromatography was equipped with a CarboPac SA-10 column (4 × 250 mm, Dionex, USA) and a pulsed amperometric detector (ED-40, Dionex, USA). The sugar analysis was conducted at 70 °C using sodium hydroxide and sodium acetate as the mobile phase at a flow rate of 0.8 mL/min. The injection volume of the sample was 10 µL. The sugar contents in the raw material and solid fraction are

Table 1

The central composite design varying on two factors: reaction time and sulfuric acid concentration with content of monomeric sugars (glucose, xylose, arabinose, galactose, and mannose) in the liquid fraction after dilute acid pretreatment.

Sample No	Run No ^a	Variables		Coded levels		Monomeric sugars (%) ^b				
		Reaction time (min)	Sulfuric acid concentration (% w/w)	Reaction time	Sulfuric acid concentration	Glucose	Xylose (Y ₁)	Arabinose	Galactose	Mannose
		X ₁	X ₂	X ₁	X ₂					
1	2	30	2	−1	−1	2.6	7.9	0.6	0.9	0.4
2	1	90	2	1	−1	4.9	15.5	0.7	1.3	0.9
3	4	30	6	−1	1	4.6	12.8	0.7	1.2	0.7
4	6	90	6	1	1	6.2	13.7	0.6	1.2	1.0
5	5	17.7	4	−1.41	0	3.2	10.3	0.7	1.0	0.5
6	8	102.3	4	1.41	0	6.5	16.0	0.7	1.3	1.0
7	7	60	1.18	0	−1.41	3.2	10.5	0.7	1.0	0.5
8	10	60	6.82	0	1.41	6.2	18.0	0.7	1.3	1.0
9 ^c	9	60	4	0	0	5.4	14.4	0.7	1.3	0.9
10 ^c	3	60	4	0	0	5.1	14.2	0.7	1.3	0.9

Reaction temperature designated at 121 °C.

^a The order of actual experiment.

^b Based on a dry weight of biomass.

^c Sample No 9 and 10 were central points.

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