pISSN: 1017-0715 eISSN: 2233-7180 http://dx.doi.org/DOI: 10.5658/WOOD.2015.43.5.578

# Organosolv Pretreatment of Slurry Composting and Biofiltration of Liquid Fertilizer-Treated Yellow Poplar for Sugar Production<sup>1</sup>

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#### **ABSTRACT**

The present study examines the influence of slurry composting and biofiltration liquid fertilizer (SCBLF) treatment on the biomass characteristics of yellow poplar, and the optimization of organosolv pretreatment for sugar production. After SCBLF treatment, total exchangeable cation contents of yellow poplar was increased from 3.1 g kg<sup>-1</sup> to 4.4 g kg<sup>-1</sup>, and as a result, biomass production of yellow poplar was also enhanced by 82.3%. Organosolv pretreatment was conducted with three independent variables: 1) reaction temperature: 133.2°C to 166.8°C; 2) acid concentration: 0.2% to 1.8%; and 3) reaction time: 1.6 min to 18.4 min. Reaction temperature was the most significant variable in water insoluble solid (WIS) recovery rate. High overall sugar yield was attained from pretreatment conditions approximately 50% of WIS recovery rate, and the highest overall glucose yield (44.0%) was achieved from pretreatment at 140°C with 1.5% acid concentration for 5 min. Consequently, 21.1% of glucose and 5.8% of xylose were produced from the organosolv pretreatment of SCBLF-treated 8-year-old yellow poplar.

**Keywords:** slurry composting and biofiltration, yellow poplar, organosolv pretreatment, biomass production, sugar production

# 1. INTRODUCTION

Excessive consumption of fossil fuels have generated considerable greenhouse gas (GHG)

emissions during the last few decades (Sarkar *et al.*, 2012). A search for new, alternative energy sources is urgently needed (Balat, 2011). Lignocellulosic biomass is recognized as the

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only suitable primary energy resource for the production of low-cost, large-amount, and high-availability energy (e.g., cellulosic ethanol or biodiesel) that can replace fossil fuels (Hamelinck *et al.*, 2005). Despite significant advantages of lignocellulosic biomass, its utilization is problematic due to its recalcitrant characteristics, which could be overcome by the development of a practicable, pretreatment process (Wyman, 2007).

Thus, many types of pretreatment processes suitable for the lignocellulosic biomass that augment fermentable sugar vield have been developed. In particular, organosolv pre-treatment has been considered a promising method for lignocellulosic biomass containing high lignin (Holtzapple and Humphrey, 1984). This pretreatment is performed using organic solvents (e.g., methanol, ethanol and acetone) in the presence of an acid or alkali catalyst to solubilize lignin and hemicellulose from the lignocellulosic biomass (Pan et al., 2006). Organosolv pretreatment can achieve a high glucose yield from a lignocellulosic biomass (Pan et al., 2007; Park et al., 2010; Koo et al., 2011). In addition, organosolv lignin extracted from the organosoly pretreatment process has a relatively high purity and diverse application (El Hage et al., 2010).

Among the lignocellulosic biomass, yellow poplar (*Liriodendron tulipifera*) is one of the most attractive rapid-growth species identified as a renewable and sustainable resource for bioenergy production. Yellow poplar has a short harvesting cycle and has great adaptability to a

variety of climatic and soil conditions. Thus, yellow poplar has been researched for sugar (Kim *et al.*, 2011) and ethanol (Koo *et al.*, 2011) production.

Slurry composting and biofiltration liquid fertilizer (SCBLF) was developed in South Korea as an alternative approach that mitigates both environmental and economic burdens of livestock excreta (Dauden and Quilez, 2004; Sanchez and Gonzalez, 2005). SCBLF is convenient to use, is clean, odorless, and homogeneous compared to other general liquid fertilizers. Hence, SCBLF has been mostly applied to crops such as rice (Kim *et al.*, 2004) and red peppers (Lim *et al.*, 2008), and recently its applications have increased to the production of lignocellulosic biomass (Lim *et al.*, 2008; Kim *et al.*, 2011).

The present study focused on the effect of SCBLF on the biomass characteristics and sugar production of yellow poplar, and the optimization of organosolv pretreatment for sugar production. First, yellow poplar was treated with SCBLF to examine the biomass characteristics, and then SCBLF-treated yellow poplar was pretreated with organosolv to improve enzymatic hydrolysis. The effects of SCBLF treatment and different pretreatment variables on sugar production were analyzed and compared. These results showed the feasibility of the SCBLF treatment and the ability of the organosolv pretreatment to maximize sugar yields.

Table 1. Characteristics of slurry composting and biofiltration liquid fertilizer

рН	Total N	Total P	K <sup>+</sup>	Ca <sup>2+</sup>	Na <sup>+</sup>	Mg <sup>2+</sup>
	(%)	(mg kg <sup>-1</sup> )				
8.3 ± 0.0	$0.1 \pm 0.0$	88.8 ± 1.7	3,204.8 ± 18.4	$45.9 \pm 0.6$	$558.7 \pm 5.4$	$10.6 \pm 0.3$

# 2. MATERIALS AND METHODS

#### 2.1. Raw materials

Five-year-old yellow poplars, grown at *Eocheon* Experimental Forest, Hwaseong-si, Gyeonggi-do, South Korea and managed by the Korea Forest Research Institute, were used for this experiment. The SCBLF was manufactured by the National Institute of Animal Science and the chemical characteristics were followed throughout the study (Table 1).

# 2.2. SCBLF treatment

To investigate the effects of SCBLF treatment on biomass characteristics and sugar production, experimental plots were divided into control and SCBLF treatment plots. After evaluating nitrogen content, we determined each tree would receive 20 ℓ per week-¹ of SCBLF for a specific period during certain years (i.e., June 2008 to October 2008; May 2009 to September 2009; June 2010 to September 2010; June 2011 to September 2011).

# 2.3. Biomass production

One tree randomly chosen from each experimental plot was cut down and its twig and stem weight were measured to confirm biomass production (May 2009, June 2010, September 2011). Next, each stem of an 8-year-old yellow poplar harvested in September 2011 was dried, ground and sieved through 0.5 mm screens and all the samples were stored at 4°C for further experiments (less than 10% of the initial moisture content).

# 2.4. Inorganic content

Exchangeable cations (Na<sup>+</sup>, Mg<sup>2+</sup>, K<sup>+</sup>, Ca<sup>2+</sup>) and heavy metals (Cr, Mn, Fe, Cu, Zn, Cd, Pb) were analyzed by an Inductively Coupled Plasma (ICP) Emission Spectrometer (ICPS-1000, Shimadzu, Japan) housed at the National Instrumentation Center for Environmental Management (Seoul, South Korea).

# 2.5. Organosolv pretreatment

Organosolv pretreatment was conducted in a 1  $\ell$  reactor (Bolted Closure Vessels, Hanul Autoclave Co. Ltd., Korea) made of stainless steel (SUS 316) and containing an inner thermocouple to control and measure the internal temperature. The reactor was loaded with 25 g of wood powder and 500 m $\ell$  of a 50:50 ethanol: water mixture (v v<sup>-1</sup>) containing sulfuric acid at concentrations from 0.2% to 1.7% (w w<sup>-1</sup>) as an acid catalyst. The reaction temperature was electrically controlled from 133 °C

to 168°C by an external controller. The reactor was preheated to the reaction temperature for 25 min to 35 min and the reaction time (from 1.6 min to 18.4 min) was monitored when the desired temperature was achieved. Afterwards, the reactor was placed in an ice chamber to cool down below room temperature. Pretreated mixtures were washed with a double volume of distilled water to remove degraded products and solvents, and divided into the solid fraction (pretreated biomass) and liquid fraction (aqueous-organosoluble fraction) by filtration using Advantec No. 2 filter paper (Toyo Roshi Kaisha Ltd., Japan). The water insoluble solid (WIS) recovery rate (Eq. (1)) was measured based on the filtered WIS weight after pretreatment, and the pretreated biomass was stored at 4°C without the drying process for enzymatic hydrolysis.

WIS recovery rate (%) =

water insoluble solid

after pretreatment (g) 
$$\times$$
 100 (1)

input amount of pretreatment (g)

The logarithm of the combined severity factor (log CS, Eq. (2)) was calculated from room temperature pH, the reaction temperature T ( $^{\circ}$ C), and reaction time t (min) without considering preheating time (Chum *et al.*, 1990):

$$\log CS = \log \left( t \times exp^{\frac{T-100}{14.75}} \right) - pH \tag{2}$$

### 2.6. Enzymatic hydrolysis

A total of 1 g of pretreated biomass (oven

dry weight) was placed in a 100 m $\ell$  working volume of 50 mol m $^{-3}$  sodium acetate buffer in a 250 m $\ell$  Erlenmeyer flask. Commercial cellulase (NS-50013, enzyme loading of 15 Filter Paper Units (FPU) g $^{-1}$  substrate) and β-glucosidase (NS-50010, 0.3 times the NS-50013 input amount), kindly provided by Novozyme Korea Ltd, were added for enzymatic hydrolysis, and incubated in a shaking incubator at 50°C, 2.5 Hz for 72 h. Enzymatic hydrolysis yield (Y<sub>1</sub>, Eq. (3)) was calculated as the percentage of released sugar yield from the pretreated biomass.

$$Y_{1} (\%) = \\ \frac{Concentration of sugar after enzymatic}{hydrolysis (g l^{-1}) \times volume (l)} \times 100$$
 (3)

In addition, to identify the productivity of sugars from the organosolv pretreatment and enzymatic hydrolysis process, the overall sugar yield based on the initial input (Y<sub>2</sub>, Eq. (4)) was calculated.

$$Y_2$$
 (%) =  $Y_1$  (%) × WIS recovery rate (%) × 100 (4)

#### 2.7. Analytical methods

The composition amount of glucan, xylan, Klason lignin and acid soluble lignin in the raw materials were analyzed according to the standard NREL LAP "Determination of Structural Carbohydrates and Lignin in Biomass" (Sluiter *et al.*, 2008). The amounts of sugars were analyzed by high performance liquid chromatography (HP 1100, Hewlett Packard, USA) at

 $40\,^{\circ}\mathrm{C}$  with 50 mol m<sup>-3</sup> sulfuric acid as the eluent at a flow rate of 0.5 m $\ell$  min<sup>-1</sup> and an injection volume of 10  $\mu\ell$ . An Aminex HPX-87H column (300 mm  $\times$  7.8 mm, 5  $\mu$ m) was used for determination of sugars and inhibitory compounds, and a refractive index detector (HP 1100, Hewlett Packard, USA) was used to quantify the products.

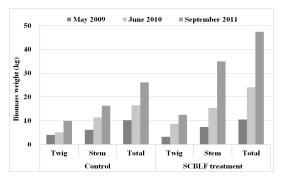
# 2.8. Data analysis

The effects of each variable were examined by the General Linear Model procedure with the Statistical Analysis System programming package (SAS ver. 9.2, SAS Institute Inc, Cary, North Carolina). A 95% confidence level was used in all statistical tests. Significant effects with p < 0.05 were further characterized by the least significant difference test between means.

# 3. RESULTS AND DISCUSSION

# 3.1. Effect of SCBLF treatment on biomass characteristics

Fig. 1 illustrates yellow poplar biomass weight measured during the SCBLF treatment period. Biomass weight significantly increased from 9.9 kg to 12.5 kg for twig, and from 16.2 kg to 34.9 kg for stem by the SCBLF treatment. In particular, the stem biomass weight was significantly increased by 115.2% in a quadrennial period of fertilizer treatment, and thus the SCBLF treatment increased total biomass production by 81.3%. In comparison to previous results that the stem biomass weight



SCBLF: slurry composting and biofiltration liquid fertilizer treatment (20  $\ell$  week<sup>-1</sup>)

Fig. 1. Biomass weight of yellow poplar on the control and SCBLF treatment.

was enhanced only 18.8% after SCBLF treatment, duration of SCBLF treatment might be a reason of enormous enhancement (Kim et al., 2012). These results are likely due to the nutrients (e.g., nitrogen, phosphorus, and potassium) in the SCBLF, which promote favorable biomass physiological conditions such as chlorophyll content or leaf area (Klooster et al., 2012). Mean leaf area and total nitrogen values in leaves were previously reported to be much higher in SCBLF-treated poplar, accordingly, the average height growth and diameter at breast height were increased 18% and 41%, respectively, by the SCBLF treatment (Lim et al., 2008). These results indicate SCBLF is an adequate fertilizer for biomass production.

Chemical composition of yellow poplar was slightly altered by the SCBLF treatment (Table 2). Glucose, xylose and total lignin contents were not significantly different between control and SCBLF-treated yellow poplar. Table 3 shows exchangeable cation contents in the yellow poplar stem. Contents of K<sup>+</sup> and Na<sup>+</sup> were

Table 2. Composition analysis of yellow poplar on the control and SCBLF treatment (based on dry weight)

	Glucose (%)	Xylose (%)	Klason lignin (%)	ASL (%)	Total lignin (%)
Control	$43.5 \pm 0.1^{(A)}$	$21.7 \pm 0.2^{(A)}$	$22.3 \pm 0.0^{(A)}$	$3.7 \pm 0.1^{(B)}$	$26.0 \pm 0.1^{(A)}$
SCBLF treatment	$43.1 \pm 0.1^{(A)}$	$21.2 \pm 0.4^{(A)}$	$21.5 \pm 0.0^{(B)}$	$4.3 \pm 0.3^{(A)}$	$25.8 \pm 0.2^{(A)}$

SCBLF: Slurry composting and biofiltration liquid fertilizer; ASL: Acid soluble lignin

Different capital letters in parenthesis indicate significant difference at p = 0.05 (least significance difference test)

Table 3. Exchangeable cation contents (g kg<sup>-1</sup>) of yellow poplar on the control and SCBLF treatment

Treatment	K <sup>+</sup>	Ca <sup>2+</sup>	Na <sup>+</sup>	$\mathrm{Mg}^{2^+}$	Total
Control	$1.4 \pm 0.1^{(B)}$	$1.1 \pm 0.1^{(A)}$	$0.2 \pm 0.0^{(B)}$	$0.4 \pm 0.0^{(A)}$	$3.1 \pm 0.2^{(B)}$
SCBLF treatment	$2.6 \pm 0.7^{(A)}$	$1.1 \pm 0.0^{(A)}$	$0.3 \pm 0.1^{(A)}$	$0.4 \pm 0.0^{(A)}$	$4.4 \; \pm \; 0.8^{\rm (A)}$

SCBLF: Slurry composting and biofiltration liquid fertilizer

Different capital letters in parenthesis indicate significant difference at p = 0.05 (least significance difference test)

significantly increased by the SCBLF treatment, whereas Ca2+ and Mg2+ were not. In SCBLF, K<sup>+</sup> and Na<sup>+</sup> contents were particularly higher than other exchangeable cation contents (Table 1), thus appearing to be effectively absorbed by the biomass. Biomass growth is positively correlated with exchangeable cation content, especially K<sup>+</sup>, in biomass and circumstance (Bilodeau-Gauthier et al., 2011; Wang and Klinka, 1997). Therefore, these results indicate that exchangeable cation contents of biomass were efficiently increased by the SCBLF treatment, and successively contributed to rapid biomass growth having different characterizations of thermal degradation features. In addition, difference of heavy metal contents in biomass and soil after SCBLF treatment were negligible (data not shown), although long-term observation is indispensable for environmentally-friendly utilization. Based on these studies, SCBLF is a preferable fertilizer for increasing yellow poplar

biomass production as a renewable energy source.

# 3.2. Enzymatic hydrolysis yield from organosolv pretreatment

WIS recovery rate ranged from 35.7% to 67.3% for the control and from 35.7% to 66.2% for the SCBLF-treated yellow poplar depending on the pretreatment conditions (Table 4). Despite a slight decline observed in SCBLF-treated yellow poplar, SCBLF treatment a statistically significant difference was not observed in the WIS recovery rate. Under the same pretreatment conditions, WIS recovery rate in this study was somewhat lower than previous results (Koo *et al.*, 2011), likely due to the different age of the raw materials. The higher reaction temperature and acid concentration significantly influenced the decrease in the WIS recovery rate. The reaction time, however, was the least

Table 4. Enzymatic hydrolysis yield of yellow poplar after organosolv pretreatment

	Pretreatment condition			WIS c recovery rate		natic <sup>d</sup> yield (Y <sub>1</sub> )	Overall <sup>e</sup> sugar yield (Y <sub>2</sub> )			
-					1000,013, 1410	Glucose	Xylose	Glucose	Xylose	Total
	(°C)	(%)	(min)	log CS b	(%)	(%)	(%)	(%)	(%)	(%)
- - - -	140	0.5	5	0.3	67.3	43.6	8.8	29.3	5.9	35.2
	160	0.5	5	0.9	46.1	92.8	6.3	42.8	2.9	45.7
	140	1.5	5	0.7	53.3	80.7	9.0	43.0	4.8	47.8
	160	1.5	5	1.3	38.1	101.1	2.5	38.5	1.0	39.5
	140	0.5	15	0.8	63.6	66.4	9.5	42.2	6.0	48.3
	160	0.5	15	1.4	41.4	101.0	4.6	41.8	1.9	43.7
-	140	1.5	15	1.2	47.9	89.1	7.5	42.7	3.6	46.3
	160	1.5	15	1.8	39.6	99.9	1.8	39.5	0.7	40.3
Control	133.2	1	10	0.7	58.0	48.3	9.3	28.0	5.4	33.4
	166.8	1	10	1.6	35.7	101.4	1.9	36.2	0.7	36.9
-	150	0.16	10	0.4	66.5	36.0	9.0	24.0	6.0	29.9
•	150	1.84	10	1.4	37.9	96.3	4.7	36.5	1.8	38.3
	150	1	1.6	0.4	45.5	83.3	9.5	37.9	4.3	42.2
-	150	1	18.4	1.4	40.8	93.0	6.0	38.0	2.4	40.4
	150	1	10	1.1	41.8	91.7	6.8	38.3	2.9	41.2
-	No pretreatment			100.0	3.4	1.6	3.4	3.4	1.6	
	140	0.5	5	0.3	65.1	52.0	9.5	33.8	6.2	40.0
•	160	0.5	5	0.9	46.1	94.2	5.8	43.5	2.7	46.1
SCBLF a treatment	140	1.5	5	0.7	52.0	84.7	9.0	44.0	4.7	48.7
	160	1.5	5	1.3	38.9	102.3	2.3	39.8	0.9	40.7
	140	0.5	15	0.8	56.6	74.3	9.4	42.1	5.3	47.4
	160	0.5	15	1.4	42.8	102.1	4.8	43.7	2.0	45.7
	140	1.5	15	1.2	49.0	86.7	6.9	42.5	3.4	45.9
	160	1.5	15	1.8	40.3	102.8	1.1	41.4	0.4	41.8
	133.2	1	10	0.7	57.3	54.6	9.6	31.3	5.5	36.8
	166.8	1	10	1.6	35.7	100.5	2.1	35.9	0.8	36.6
	150	0.16	10	0.4	66.2	42.6	10.0	28.2	6.6	34.8
	150	1.84	10	1.4	38.6	101.9	5.1	39.3	2.0	41.2
	150	1	1.6	0.4	50.5	81.6	10.0	41.2	5.1	46.3
	150	1	18.4	1.4	42.2	93.1	6.1	39.3	2.6	41.8
	150	1	10	1.1	42.5	90.7	7.3	38.6	3.1	41.7
		No pr	etreatmer	nt	100.0	4.2	2.0	4.2	4.2	2.0

affected. The reactor used in this study required approximately 30 min to reach the desired reaction temperature, thus the preheating time possibly diminished the effect of reaction time.

Interestingly, the lowest WIS recovery rate was achieved from pretreatment at 166.8% with 1% acid concentration for 10 min (log CS: 1.6), and not at 160% with 1.5% acid concentration

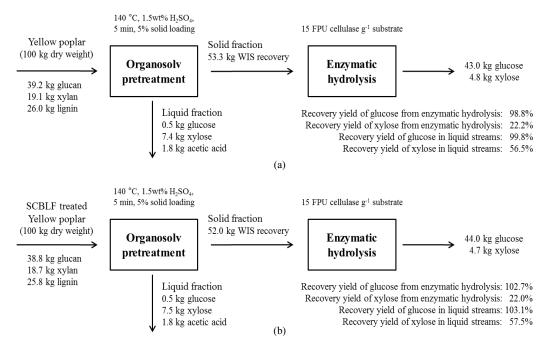
for 15 min (log CS: 1.8), which is a more severe pretreatment condition. This result was also found in several other pretreatment conditions (e.g. between 140°C, 1.5% acid, 5 min (log CS: 0.7) and 140°C, 0.5% acid, 15 min (log CS: 0.8); between  $160^{\circ}$ C, 1.5% acid, 5 min (log CS: 1.3) and 160°C, 0.5% acid, 15 min (log CS: 1.4)). Similar results showed a higher degradation rate with the low logarithm of the combined severity factor (Shi et al., 2011). These results are potentially due to the reaction temperature and acid concentration affecting WIS recovery rate more than the reaction time. As a result, many researchers use the log CS as the indicator of energy cost or pretreatment efficiency (Goh et al., 2011). But to be used for that purpose, improvement of the equation may be necessary.

Enzymatic hydrolysis yield of the control and SCBLF-treated yellow poplar are shown in Table 4. Similar to WIS recovery rate, SCBLF treatment did not significantly influence the enzymatic hydrolysis yield, despite being slightly higher in SCBLF-treated yellow poplar. WIS recovery rate of both control and SCBLF-treated yellow poplar, pretreated at 140°C, with 0.5% acid concentration for 15 min was similar to pretreatment at 133.2°C, with 1% acid concentration for 10 min, but enzymatic hydrolysis yield was significantly enhanced. This result confirmed that reaction temperature was a more important variable than acid concentration or reaction time as described earlier. In comparison to previous results that approximately 80% monomeric sugar was yielded from organosolv

pretreatment at 150°C with an alkali catalyst, the higher enzymatic hydrolysis yield was obtained with the lower reaction temperature and catalyst concentration (Koo et al., 2011). Additionally, in dilute acid pretreatment, enzymatic hydrolysis yield of glucose was approximately 85% at a log CS of 2.6, despite using rice straw as a raw material (Hsu et al., 2010). In the present study, a high enzymatic hydrolysis yield of glucose over 100% was obtained from pretreatment conditions at a log CS of 1.3. In summary, organosoly pretreatment with an acid catalyst is more effective and economic than organosoly pretreatment with an alkali catalyst or dilute acid pretreatment, because of advantages such as simultaneous solubilization of lignin and hemicelluloses (Kumar et al., 2009).

# 3.3. Overall sugar yield

Enzymatic hydrolysis yield of glucose and WIS recovery rate were mostly in an inverse proportion to each other. In other words, enzymatic hydrolysis yield of glucose was enhanced with the decrease of WIS recovery rate, when reaction temperature, acid concentration, and reaction time were increased. Thus, the overall sugar yield from organosolv pretreatment and enzymatic hydrolysis was calculated (Table 4). The highest overall glucose yield (44.0%) was achieved from pretreatment of SCBLF-treated yellow poplar at 140°C with 1.5% acid concentration for 5 min (log CS: 0.7). Higher overall glucose yield was attained from the early 50%



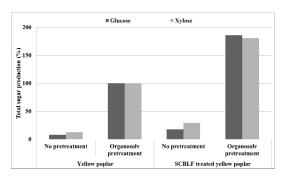
Recovery yield refers to the recovery rate of sugars based on the sugar content in the raw materials WIS recovery: water-insoluble solid recovery

SCBLF: slurry composting and biofiltration liquid fertilizer treatment (20 ℓ week⁻¹)

Fig. 2. Sugar balance for organosolv pretreatment and enzymatic hydrolysis of yellow poplar on the control (a) and SCBLF treatment (b).

of WIS recovery rate, irrespective of SCBLF treatment. From this point, overall glucose yield declined because the decrease rate of WIS recovery rate was much higher than the increase rate of enzymatic hydrolysis yield. Previous researches reported that SCBLF treated yellow poplar could be utilized to produce bioethanol, however pretreatment conditions applied to those researches were somewhat severe to obtain maximum yield (Kim *et al.*, 2011; Kim *et al.*, 2012). In comparison to previous studies, in this study tried to find out optimal condition for sugar production and it was obtained with low value of log CS than

that of previous studies (150°C with 1% acid concentration for 10 min (log CS: 1.1). These results might be occurred because of using juvenile lignocellulosic biomass which is less lignified. Compared to the dilute acid pretreatment, similarly, the maximum overall sugar yield was not augmented continuously, however log CS values (between 2.2 and 2.4) achieving the maximum overall sugar yield were 3 times higher than with organosoly pretreatment (Hsu *et al.*, 2010; Jensen *et al.*, 2010). These results probably related to complex and/or individual degradation aspects of lignocellulosic biomass major components, and further research



Based on the sugar production after organosolv pretreatment of yellow poplar

Reflect the actual biomass production of yellow poplar by the SCBLF treatment (control: 26.12 kg, SCBLF treatment: 47.36 kg) SCBLF: slurry composting and biofiltration liquid fertilizer treatment (20  $\ell$  week<sup>-1</sup>)

Fig. 3. Total sugar production of yellow poplar on the control and SCBLF.

is required to reveal fundamental characteristics of lignocellulosic biomass and design pretreatment process much economically.

Fig. 2 summarizes sugar balance for organosolv pretreatment conditions of the highest overall sugar yield of control and SCBLF-treated yellow poplar. On the basis of 100 kg of raw material, approximately 52 kg to 53 kg of pretreated solid was recovered, and almost all glucan was hydrolyzed by enzymatic hydrolysis (control: 43.00 kg; SCBLF treatment: 44.0 kg). Recovery yield of xylose from enzymatic hydrolysis (control: 22.2%; SCBLF treatment: 22.0%) was higher than other acid-based pretreatments, because the pretreatment conditions in the present study were lower than other pretreatments and led to a partial amount of xylan remaining in the pretreated biomass. However, for the same reason, recovery yield of xylose in liquid streams was reduced due to a relatively small amount of xylose in the liquid fraction of organosolv pretreatment.

# 3.4. Total sugar production

Fig. 3 shows total sugar production of yellow poplar by the SCBLF treatment and organosolv pretreatment. Total biomass production (81.3%) was enhanced by the SCBLF treatment and overall sugar production was maximized by organosolv pretreatment. Based on the sugar production after organosoly pretreatment of yellow poplar, total production of glucose and xylose were 85.7% and 80.6% increased after 4 years of SCBLF treatment. Total xylose production was lower than previous results with dilute acid pretreatment because of the mild pretreatment condition (Hsu et al., 2010; Jensen et al., 2010). Conversely, as an advantage, a large amount of xylose can be comfortably recovered from the enzymatic hydrolysis process, and the other xylose component easily utilized through a mild detoxification process, because of the low amount of inhibitory compounds in the liquid fraction after pretreatment (acetic acid: less than 1 g L-1; 5-HMF and furfural: not detected). Consequently, combination of the SCBLF treatment and organosolv pretreatment enhanced not only biomass production but also the pretreatment efficiency, thus maximizing sugar production.

### 4. CONCLUSION

Biomass production of yellow poplar was significantly enhanced by the SCBLF treatment, and the highest overall glucose yield (44.0%) was achieved from organosolv pretreatment of SCBLF-treated yellow poplar at 140°C with 1.5% acid concentration for 5 min (log CS: 0.7). A total of 21.08 kg of glucose and 5.8 kg of xylose were produced from a 5-year-old yellow poplar after 3 years of SCBLF treatment. Application of SCBLF to lignocellulosic biomass drastically mitigated disposal cost of livestock excreta and augmented biomass production, but long-term observation of the influence of SCBLF on plant physiology and the environment is required.

# **ACKNOWLEDGMENTS**

This work was carried out with the support of a "Forest Science & Technology Projects (Project No. S120811L140130)" grant provided by the Korea Forest Service and "Cooperative Research Program for Agriculture Science & Technology Development (Project No. 20110401-030-568-001-04-00)" grant provided by Rural Development Administration, Republic of Korea.

# **REFERENCES**

- Balat, M. 2011. Production of bioethanol from lignocellulosic materials via the biochemical pathway:

  A review. Energy Conversion and Management 52(2): 858~875.
- Bilodeau-Gauthier, S., Houle, D., Gagnon, C., Côté, B., Messier, C. 2011. Assessment of sugar maple tree growth in relation to the partitioning of elements in xylem along a soil acidity gradient. Forest Ecology and Management 261(1): 95~104.

- Chum, H.L., Johnson, D.K., Black, S.K., Overend, R.P. 1990. Pretreatment-catalyst effects and the combined severity parameter. Applied Biochemistry and Biotechnology 24(1): 1~14.
- Dauden, A., Quilez, D. 2004. Pig slurry versus mineral fertilization on corn yield and nitrate leaching in a Mediterranean irrigated environment. European Journal of Agronomy 21(1): 7~19.
- El Hage, R., Chrusciel, L., Desharnais, L., Brosse, N. 2010. Effect of autohydrolysis of *Miscanthus x giganteus* on lignin structure and organosolv delignification. Bioresource Technology 101(23): 9321~9329.
- Goh, C.S., Tan, H.T., Lee, K.T., Brosse, N. 2011. Evaluation and optimization of organosolv pretreatment using combined severity factors and response surface methodology. Biomass & Bioenergy 35(9): 4025~4033.
- Hamelinck, C.N., Hooijdonk, G., Faaij, A.P.C. 2005. Ethanol from lignocellulosic biomass: techno-economic performance in short-, middle-and long-term. Biomass & Bioenergy 28(4): 384~410.
- Holtzapple, M., Humphrey, A. 1984. The effect of organosolv pretreatment on the enzymatic hydrolysis of poplar. Biotechnology and Bioengineering 26(7): 670~676.
- Hsu, T.C., Guo, G.L., Chen, W.H., Hwang, W.S. 2010. Effect of dilute acid pretreatment of rice straw on structural properties and enzymatic hydrolysis. Bioresource Technology 101(13): 4907~4913.
- Jensen, J.R., Morinelly, J.E., Gossen, K.R., Brodeur-Campbell, M.J., Shonnard, D.R. 2010. Effects of dilute acid pretreatment conditions on enzymatic hydrolysis monomer and oligomer sugar yields for aspen, balsam, and switchgrass. Bioresource Technology 101(7): 2317~2325.
- Kim, H.C., Yeo, J.K., Koo, Y.B., Shin, H.N., Choi,

- J.Y., Lee, H.H. 2011. Growth and Biomass Production of Fast Growing Tree Species Treated with Slurry Composting and Biofiltration Liquid Fertilizer. Korean Journal of Soil Science and Fertilizer 44(2): 206~214.
- Kim, H.Y., Gwak, K.S., Kim, H.Y., Ryu, K.O., Kim, P.G., Cho, D.H., Choi, J.Y., Choi, I.G. 2011. Effect of Treatment Amounts of Slurry Composting and Biofiltration Liquid Fertilizer on Growth Characteristics and Biothanol Production of Yellow Poplar. Journal of the Korean Wood Science and Technology 39(6): 459~468.
- Kim, H.Y., Gwak, K.S., Lee, S.Y., Jeong, H.S., Ryu, K.O., Choi, I.G. 2012. Biomass characteristics and ethanol production of yellow poplar (*Liriodendron tulipifera*) treated with slurry composting and biofiltration liquid as fertilizer. Biomass & Bioenergy 42: 10~17.
- Kim, J.S., Lee, K.B., Lee, D.B., Lee, S.B., Na., S.Y. 2004. Influence of liquid pig manure on rice growth and nutrient movement in paddy soil under different drainage conditions. Korean Journal of Soil Science and Fertilizer 37: 97~103.
- Klooster, W.S., Cregg, B.M., Fernandez, R.T., Nzokou, P. 2012. Growth and physiology of deciduous shade trees in response to controlled-release fertilizer. Scientia Horticulturae 135: 71~79.
- Koo, B.W., Kim, H.Y., Park, N., Lee, S.M., Yeo, H., Choi, I.G. 2011. Organosolv pretreatment of *Liriodendron tulipifera* and simultaneous saccharification and fermentation for bioethanol production. Biomass & Bioenergy 35(5): 1833~ 1840.
- Koo, B.W., Park, N., Jeong, H.S., Choi, J.W., Yeo, H., Choi, I.G. 2011. Characterization of by-products from organosolv pretreatments of yellow poplar wood (*Liriodendron tulipifera*) in the presence of acid and alkali catalysts. Journal

- of Industrial and Engineering Chemistry 17(1):  $18 \sim 24$ .
- Kumar, P., Barrett, D.M., Delwiche, M.J., Stroeve, P. 2009. Methods for pretreatment of lignocellulosic biomass for efficient hydrolysis and biofuel production. Industrial & Engineering Chemistry Research 48(8): 3713~3729.
- Lim, T.J., Hong, S.D., Kim, S.H., Park, J.M. 2008. Evaluation of Yield and Quality from Red Pepper for Application Rates of Pig Slurry Composting Biofiltration. Korean Journal of Environmental Agriculture 27(2): 171~177.
- Pan, X., Gilkes, N., Kadla, J., Pye, K., Saka, S., Gregg, D., Ehara, K., Xie, D., Lam, D., Saddler, J. 2006. Bioconversion of hybrid poplar to ethanol and co-products using an organosolv fractionation process: Optimization of process yields. Biotechnology and Bioengineering 94(5): 851~861.
- Pan, X., Xie, D., Yu, R., Lam, D., Saddler, J. 2007.
  Pretreatment of lodgepole pine killed by mountain pine beetle using the ethanol organosolv process: Fractionation and process optimization.
  Industrial & Engineering Chemistry Research 46(8): 2609~2617.
- Park, N., Kim, H.Y., Koo, B.W., Yeo, H., Choi, I.G. 2010. Organosolv pretreatment with various catalysts for enhancing enzymatic hydrolysis of pitch pine (*Pinus rigida*). Bioresource Technology 101(18): 7046~7053.
- Sarkar, N., Ghosh, S.K., Bannerjee, S., Aikat, K. 2012. Bioethanol production from agricultural wastes: An overview. Renewable Energy 37(1):  $19\sim27$ .
- Sanchez, M., Gonzalez, J. 2005. The fertilizer value of pig slurry. I. Values depending on the type of operation. Bioresource Technology 96(10): 1117~1123.
- Shi, J., Ebrik, M.A., Wyman, C.E. 2011. Sugar

yields from dilute sulfuric acid and sulfur dioxide pretreatments and subsequent enzymatic hydrolysis of switchgrass. Bioresource Technology 102(19): 8930~8938.

Sluiter, A., Hames, B., Ruiz, R., Scarlata, C., Sluiter, J., Templeton, D., Crocker, D. 2008. Determination of structural carbohydrates and lignin in biomass. Laboratory Analytical Procedure (LAP), NREL/TP-510-42618, Revised April 2008.

- Wang, G., Klinka, K. 1997. White spruce foliar nutrient concentrations in relation to tree growth and soil nutrient amounts. Forest Ecology and Management 98(1): 89~99.
- Wyman, C.E. 2007. What is (and is not) vital to advancing cellulosic ethanol. Trends in Biotechnology 25(4): 153~157.