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Optimization of chip size and moisture content to obtain high, combined sugar recovery after sulfur dioxide-catalyzed steam pretreatment of softwood and enzymatic hydrolysis of the cellulosic component

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HIGHLIGHTS

- We steam pretreated softwood chips and then used the solids for enzymatic hydrolysis.
- We report on the influences of chip size and moisture content on sugar recovery.
- Optimizing chip size provides a small increase in sugar recovery after pretreatment.
- Elevated chip moisture provides maximum sugar recovery after both process steps.
- Elevated chip moisture also promotes good process control of the pretreatment step.

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ABSTRACT

The influence of chip size and moisture content on the combined sugar recovery after steam pretreatment of lodgepole pine and subsequent enzymatic hydrolysis of the cellulosic component were investigated using response surface methodology. Chip size had little influence on sugar recovery after both steam pretreatment and enzymatic hydrolysis. In contrast, the moisture of the chips greatly influenced the relative severity of steam pretreatment and, as a result, the combined sugar recovery from the hemicellulosic and cellulosic fractions. Irrespective of chip size and the pretreatment temperature, time, and SO_2 loading that were used, the relative severity of pretreatment was highest at a moisture of 30–40 w/w%. However, the predictive model indicated that an elevated moisture content of roughly 50 w/w% (about the moisture content of a standard softwood mill chip) would result in the highest, combined sugar recovery (80%) over the widest range of steam pretreatment conditions.

1. Introduction

Renewable fuels and chemicals can be produced from the sugars derived from the hemicellulosic and cellulosic components of biomass after pretreatment, enzymatic hydrolysis and fermentation. Concerns over volatile oil prices, energy security, the environmental consequences of continued fossil fuel dependence and the limited supplies of sugar and starch rich biomass for both human consumption and conversion to ethanol have encouraged ongoing work into the development of biomass-to-sugars-and-fuels/chemicals processes (NREL, 2008; Representative Rahall NJ II, 2007; USDA, 2011). Agricultural residues (such as corn stover and wheat straw), herbaceous energy crops (such as switchgrass) and forest residues (such as softwood and hardwood chips) have all been investigated as potential feedstocks for bioconversion to fuels and chemicals (Galbe and Zacchi, 2012). A fairly recent Canadian estimate placed the total national available biomass at between 24 and 87 million dry tonnes per year, of which forest residues may contribute as much as 80% (Mabee and Saddler, 2010). A more recent assessment concluded that the availability of forest residues in British Columbia (primarily softwood) would be sufficient to support up to 10 bioconversion facilities for the production of advanced bioethanol (Mabee et al., 2011).

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Pretreatment is widely recognized as a necessary first step in the bioconversion process, although many different strategies for the pretreatment of lignocellulosic biomass have been proposed (Chandra et al., 2007; Galbe and Zacchi, 2007; Yang and Wyman, 2008). A variation of steam pretreatment is arguably the preferred method of pretreatment as it appears to be the technology of choice for a number of advanced bioethanol facilities in Europe (Inbicon, Chemtex), North America (Abengoa, DSM-POET) and Brazil (Raizen, GranBio). However, all of these facilities primarily utilize herbaceous biomass feedstocks. If softwoods are to be used, earlier work has indicated that an acid catalyst needs to be combined with steam pretreatment to both enhance subsequent enzymatic hydrolysis of the cellulosic component while providing better recovery of the hemicellulose derived sugars (Boussaid et al., 1998; Clark and Mackie, 1987). Although previous work has shown that the addition of SO_2 did not result in significant sulfonation or the solubilization of the lignin it did appear to increase the accessibility of the enzymes to the cellulose, possibly through the modification and redistribution of the lignin fraction (Clark et al., 1989; Donaldson et al., 1988). This work also showed that, when using softwoods, more severe conditions are required to produce a cellulosic fraction that can be readily hydrolyzed at low enzyme loadings. However, at these more severe conditions a significant amount of the hemicellulose derived sugars are degraded. Thus, to try to achieve maximum combined sugar recovery (from both the hemicellulose and cellulose components) the steam pretreatment conditions have to be a compromise between maximizing the recovery of hemicellulose-derived sugars and enhancing the enzymatic hydrolysis of the cellulosic component.

Several organic acids, and sulfuric acid (H_2SO_4) in particular, have been proposed as effective acid catalysts (Lee and Jeffries, 2011; Galbe and Zacchi, 2002). As another acid catalyst, sulfur dioxide (SO_2) has been shown to have several advantages such as its rapid penetration into and its uniform distribution throughout wood chips (Mamers and Menz, 1984; Canadian Pulp and Paper Association, 1985; Wayman et al., 1984). Although less attention has been paid to the initial size and moisture content of the lignocellulosic feedstock, these two parameters are known to influence the effectiveness of steam pretreatment (Brownell et al., 1986; Monavari et al., 2009; Ballesteros et al., 2000; Ewanick and Bura, 2011). For example, the relative severity of steam pretreatment has been shown to decrease as the size of the softwood feedstock increases (Cullis et al., 2004). In subsequent work Swedish researchers were able to increase the combined recovery of soluble glucose and mannose after the pretreatment and enzymatic hydrolysis of softwood, from 71% to 73%, by decreasing chip thickness from 5–6 mm to 1–2 mm (Monavari et al., 2009). Earlier work using aspen (*Populus tremuloides* Michaux) showed that the interior temperature of green chips increased more slowly than did that of air dried chips during steam pretreatment. This difference in temperature profile resulted in the 'undercooking' of chip interiors and the 'overcooking' of chip exteriors at high reaction temperatures (Brownell et al., 1986). In other work the residual xylan content of the pretreatment-derived solid fraction of switchgrass decreased, from 3.9 to 2.5 w/w% (weight percent), as

Table 1
Experimental design for the response surface methodology model of lodgepole pine.

Run	Temperature (°C)	Time (min)	SO_2 (w/w%)	Chip size (inch)	Moisture content (w/w%)
1[a]	195	2.75	1.5	3/8	22.5
4	215	7.25	1.5	3/8	22.5
6	215	2.75	3.5	3/8	22.5
7	195	7.25	3.5	3/8	22.5
10	215	2.75	1.5	7/8	22.5
11	195	7.25	1.5	7/8	22.5
13	195	2.75	3.5	7/8	22.5
16	215	7.25	3.5	7/8	22.5
18	215	2.75	1.5	3/8	47.5
19	195	7.25	1.5	3/8	47.5
21	195	2.75	3.5	3/8	47.5
24	215	7.25	3.5	3/8	47.5
25	195	2.75	1.5	7/8	47.5
28	215	7.25	1.5	7/8	47.5
30	215	2.75	3.5	7/8	47.5
31	195	7.25	3.5	7/8	47.5
33[b]	205	5	2.5	5/8	35
34	205	5	2.5	5/8	35
35	205	5	2.5	5/8	35
36	205	5	2.5	5/8	35
37	205	5	2.5	5/8	35
38	205	5	2.5	5/8	35
39[c]	185	5	2.5	5/8	35
40	225	5	2.5	5/8	35
41	205	0.5	2.5	5/8	35
42	205	9.5	2.5	5/8	35
43	205	5	0.5	5/8	35
44	205	5	4.5	5/8	35
45	205	5	2.5	1/8	35
46	205	5	2.5	9/8	35
47	205	5	2.5	5/8	10
48	205	5	2.5	5/8	60

[a] Samples 1–31 belong to the fractional factorial design.
[b] Samples 33–38 are replicates of the center point.
[c] Samples 39–48 are axial points.

Table 2
Summary of results for steam pretreated lodgepole pine: water insoluble fraction.

Run	WIF yield	WIF composition				Glucan hydrolysis[e]
		AIL[a]	Glu[b]	Man[c]	Xyl[d]	
(–)	(w/w%)[f]	(w/w WIF%)[g]				(%)
1	56.9	50.09	49.96	0.51	0.49	38.1
4	47.6	72.66	24.03	0.15	0.11	75.1
6	47.3	68.17	32.50	0.25	0.08	66.5
7	54.3	56.15	42.55	0.29	0.17	49.7
10	56.3	48.34	48.63	0.39	0.29	40.3
11	57.4	52.50	44.05	0.34	0.26	44.9
13	59.7	47.35	51.90	0.45	0.36	32.5
16	49.9	85.85	10.98	0.16	0.14	88.1
18	52.1	51.31	48.86	0.31	0.26	44.5
19	59.9	43.84	55.31	0.32	0.26	27.8
21	61.0	40.74	56.98	0.55	0.60	24.3
24	44.8	74.23	23.77	0.00	0.00	76.8
25	62.0	42.83	57.39	0.49	0.58	22.5
28	50.2	62.48	35.03	0.32	0.24	61.7
30	49.8	57.11	43.14	0.34	0.24	53.2
31	56.5	48.79	50.90	0.26	0.18	37.4
CP[h]	49.6	60.30	38.38	0.28	0.22	58.4
SE[i]	3.4	6.77	12.13	29.50	34.41	11.1
39	61.2	42.54	56.68	0.60	0.59	24.5
40	41.9	88.40	9.89	0.00	0.00	91.0
41	65.8	38.84	58.26	0.92	1.01	16.5
42	49.6	61.81	36.55	0.23	0.21	60.5
43	59.3	46.36	53.05	0.42	0.31	31.5
44	47.7	61.53	35.53	0.18	0.11	63.1
45	51.2	57.62	41.32	0.22	0.07	53.9
46	49.0	53.19	43.25	0.29	0.12	53.8
47	62.9	46.71	52.18	0.54	0.52	28.5
48	58.9	45.43	55.46	0.47	0.38	28.8

[a] Acid insoluble lignin.
[b] Glucan.
[c] Mannan.
[d] Xylan.
[e] Calculated as the glucan present in the raw wood minus that remaining in the WIF after steam pretreatment.
[f] Expressed in units of w/w% of raw wood.
[g] Expressed in units of odg w/w% of the water insoluble fraction.
[h] Samples 33–38 are replicates of the center point.
[i] Standard error of the center point.