Size effect on Mild bisulfite pretreatment performance on Douglas fir

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Introduction

Biofuel production from biomass is a promising way to build a new fuel supply system in the future. The current pulp mill has a type of continuous digester which can handle with fine material such as sawdust and wood chip screenings from forestry industry. Using fine material with current pulp mill equipment will be a cost effective was to implement new biofuel development. However, size reduction plays an important role in the biofuel production chain through affecting pretreatment efficiency and enzymatic hydrolysis (Vidal, Dien et al. 2011; Zhang, Zhang et al. 2013). Also, size reduction is an energy intensive process, especially when using wooden material as feedstock (Bitra, Womac et al. 2009; Repellin, Govin et al. 2010; Miao, Grift et al. 2011). High energy consumption of size reduction increased the cost of biofuel production, which lowering the application possibility of new fuels. Therefore, how to compromise the initial particle size of biomass with high final product yield is what we are interested in.

Objective

The objective of this present study is to investigate the size effects on mild bisulfite pretreatment performance through characterization of pretreated solid and liquid by composition analysis, XRD and FTIR analysis. Also, temperature was include as a variable in pretreatment condition to better analyze size effects on pretreatment.

Methods

- Material & Equipment
  - I. Four initial sizes of Douglas fir were used in this study: Chip, 7/16”, 1/4”, 1/16”
  - II. 1L Parr reactor
  - III. Chemical loading: Calcium bisulfite: 6.4% ODW wood, Free SO_2 : 2.0% ODW wood
- Pretreatment process
  - I. Liquid to wood (ODW) ratio: 4:1, 60g ODW wood/batch, 240g liquor/batch
  - II. Pretreatment temperature: 135ºC, 145ºC, 155ºC; time duration: 3h
  - III. After pretreatment, vacuum filtration was performed, liquor was collected and material was washed with 1L pure water
  - IV. Pretreated solid & liquor were stored in -20ºC freezer until analysis

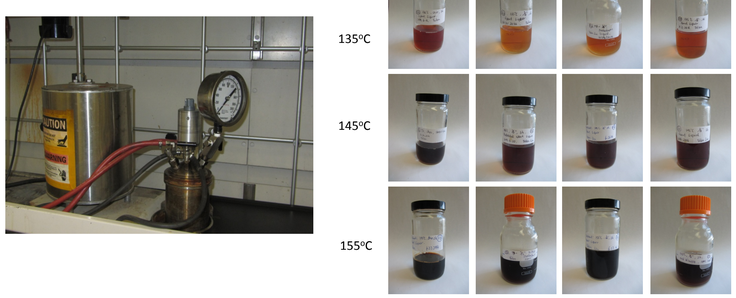

FIGURE 1-1 1L Parr reactor (Left) and pretreated liquor (Right).

Solid recovery of pretreated material

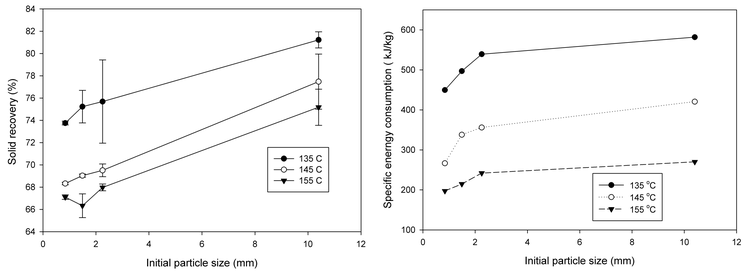

FIGURE 2-1 Solid recovery (Left) and specific energy consumption milling to 60 mesh with Wiley mill (Right) of pretreated material at various temperature and initial size.

Pretreated solid composition analysis

TABLE 1 Compositional analysis of pretreated solid.

Run No.	Temperature (ºC)	Size	Arabinan	Galactan	Glucan	Xyl/Mann	ASL	AIL	Total
	-	Untreated wood	1.4%	3.2%	48.3%	16.9%	2.0%	25.3%	97.2%
F7	135	Chip	0.2%	0.6%	50.5%	4.8%	5.3%	32.4%	93.9%
F12	135	7/16"	0.0%	0.1%	52.5%	5.3%	3.8%	32.3%	93.9%
F4	135	1/4"	0.0%	0.5%	56.1%	5.7%	3.4%	31.2%	96.9%
F10	135	1/16"	0.0%	0.0%	54.5%	4.7%	3.0%	32.2%	94.3%
F6	145	Chip	0.1%	0.5%	54.8%	3.4%	3.4%	32.2%	94.4%
F8	145	7/16"	0.0%	0.0%	58.4%	2.0%	3.3%	33.6%	97.3%
F16	145	1/4"	0.0%	0.0%	60.0%	2.2%	3.1%	33.7%	99.0%
F5	145	1/16"	0.0%	0.0%	57.5%	1.6%	2.3%	35.4%	96.8%
F17	155	Chip	0.0%	0.2%	56.1%	1.7%	5.1%	35.0%	98.2%
F1	155	7/16"	0.0%	0.0%	58.0%	1.1%	2.7%	36.2%	98.1%
F19	155	1/4"	0.0%	0.0%	51.2%	0.8%	3.9%	37.3%	93.1%
F14	155	1/16"	0.0%	0.0%	51.0%	0.8%	3.1%	40.2%	95.2%

- Hemicellulose removal was increasing along with decrease of particle and increase of treatment temperature.

Pretreated liquor composition analysis

Table 2 Pretreated liquor composition analysis.

Run No.	Temperature (ºC)	Size	Sugar recovered in solid (g)	Sugar in pretreated liquor (g)					Total Sugar recovery
				Arabinan	Galactan	Glucan	Xyl/Man	Total	
F7	135	Chip	21.53	0.63	1.56	1.78	6.84	10.81	92.5%
F12	135	7/16"	26.53	0.68	1.72	1.75	7.53	11.68	91.1%
F4	135	1/4"	28.14	0.68	1.81	1.82	7.71	12.02	95.8%
F10	135	1/16"	26.07	0.69	1.75	2.16	7.98	12.57	92.1%
F6	145	Chip	21.09	0.56	1.34	2.21	7.40	11.50	93.3%
F8	145	7/16"	24.71	0.66	1.67	2.86	8.62	13.80	91.8%
F16	145	1/4"	25.60	0.58	1.51	2.63	7.83	12.54	90.9%
F5	145	1/16"	24.19	0.57	1.52	2.89	8.19	13.17	89.1%
F17	155	Chip	20.71	0.39	0.99	2.42	5.17	8.97	84.9%
F1	155	7/16"	24.03	0.46	1.37	3.29	6.70	11.82	85.5%
F19	155	1/4"	20.81	0.42	1.25	3.97	6.01	11.66	77.4%
F14	155	1/16"	20.78	0.38	1.16	3.49	5.58	10.62	74.9%

- Total sugar recovery decrease as a decrease of particle size and increase of treatment temperature.

XRD analysis of pretreated solid

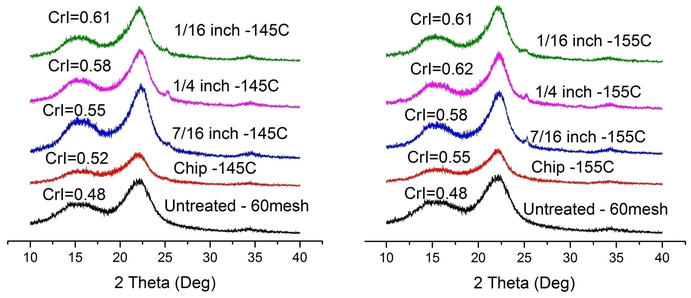

FIGURE 2-2 XRD spectra of pretreated solid milled to 60mesh.

FTIR Analysis of pretreated solid

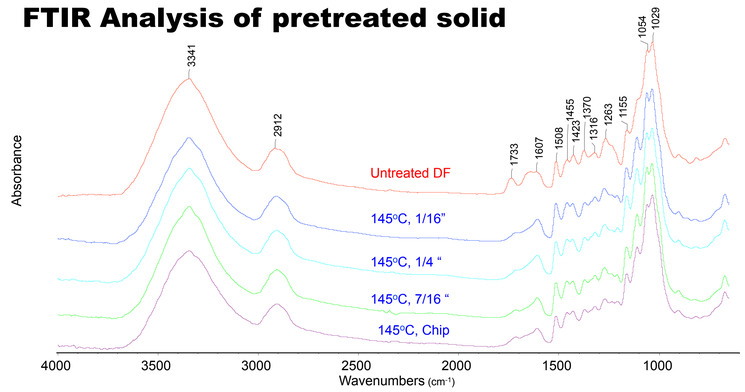

FIGURE 2-3 FTIR spectra of pretreated solid under various conditions.

Conclusions

- I. Solid recovery decreased along with a decrease of initial particle size and a increase of temperature.
- II. Under same treatment condition, hemi-cellulose was removed to a large extent at smaller particle size, however excessive reduction of size also increased some extent removal of cellulose.
- III. The total sugar recovered form solid and liquor decreased as a decrease of initial particle size at 145ºC and 155ºC. Small particle size and high treatment temperature resulted in more degradation products during pretreatment.
- IV. Crystallinity of pretreated solid all increased comparing to untreated material, CrI increased along with a decrease of initial particle size at 145ºC and 155oC, however at 135ºC the difference of CrI between different initial sizes is small (0.49-0.51).
- V. FTIR analysis also showed the removal of hemi-cellulose and structure changes of lignin.

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