

Hydrothermal alkaline sulfite pretreatment in the delivery of fermentable sugars from sugarcane bagasse

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Table 1. The pH of the reaction solution between and after pretreatment

Run	Pretreatment conditions			pH	
	S (wt.%)	T (°C)	t (min)	before	after
				pretreatment	
1	10	140	30	12.6	8.5
2	10	140	45	12.7	9.0
3	10	140	60	12.5	8.8
4	10	150	30	12.6	8.6
5	10	150	45	12.8	9.0
6	10	150	60	12.6	8.9
7	10	160	30	12.6	8.9
8	10	160	45	12.8	8.9
9	10	160	60	12.7	9.0
10	7.5	140	30	12.5	8.4
11	7.5	140	45	12.7	8.5
12	7.5	140	60	12.7	9.0
13	7.5	150	30	12.6	8.9
14	7.5	150	45	12.7	8.9
15	7.5	150	60	12.7	8.9
16	7.5	160	30	12.8	8.9
17	7.5	160	45	12.7	8.7
18	7.5	160	60	12.6	8.5
19	5	140	30	12.7	8.9
20	5	140	45	13.0	8.9
21	5	140	60	12.8	8.9
22	5	150	30	13.1	8.6
23	5	150	45	12.8	8.6
24	5	150	60	13.0	8.6
25	5	160	30	12.9	8.7
26	5	160	45	13.0	8.5
27	5	160	60	12.8	8.2

Table 2. The enzymatic glucan to glucose or xylan to xylose hydrolysis yields (mol.%) of solids produced under various conditions of HAS pretreatment.

Run	Pretreatment conditions			Glucan to glucose yield (mol.%)						Xylan to xylose yield (mol.%)					
	S (wt.%)	T (°C)	t (min)	2h	4h	8h	24h	48h	72h	2h	4h	8h	24h	48h	72h
1	10	140	30	7.7±0.1	21.3±0.6	31.5±1.9	47.9±1.9	59.0±3.1	64.7±4.4	5.4±0.0	15.9±0.1	24.6±1.2	40.2±1.9	51.9±1.4	58.2±4.2
2	10	140	45	7.7±0.2	19.5±1.7	33.5±1.0	48.1±0.7	56.9±2.2	66.2±0.4	5.5±0.1	14.6±1.1	26.7±1.6	39.7±1.1	49.2±2.0	58.5±0.9
3	10	140	60	6.3±0.5	17.6±0.8	27.4±2.1	47.0±3.6	56.5±3.0	70.9±2.7	4.7±0.4	14.4±0.8	23.8±1.2	43.6±5.6	52.7±3.4	63.7±9.1
4	10	150	30	7.8±0.0	20.7±1.1	30.6±0.2	53.9±0.4	63.3±1.2	72.0±0.8	5.3±0.1	16.3±0.5	25.7±0.0	47.4±0.6	57.8±0.0	66.5±0.3
5	10	150	45	7.5±0.6	19.8±0.7	31.6±1.1	53.5±4.4	67.7±0.8	78.4±4.5	5.3±0.3	15.4±0.7	27.0±1.5	46.2±3.9	59.3±0.0	69.9±3.9
6	10	150	60	15.8±1.1	22.6±3.4	31.8±3.3	56.1±5.2	69.2±1.3	73.8±5.1	11.8±0.9	19.7±3.2	28.2±3.5	48.6±5.0	60.7±0.1	66.1±5.1
7	10	160	30	12.6±0.7	21.5±1.3	31.3±0.4	54.7±1.8	68.4±1.5	74.2±0.8	10.4±0.4	19.6±1.1	28.2±0.3	47.7±1.3	60.0±1.0	66.6±0.7
8	10	160	45	13.6±3.4	22.9±2.5	35.7±1.6	56.3±1.8	73.9±3.4	79.9±1.6	11.4±2.7	20.3±1.9	31.9±2.7	46.9±1.7	62.3±3.8	70.7±2.6
9	10	160	60	15.5±0.9	21.8±0.8	32.2±0.1	54.1±2.4	69.4±0.3	75.6±0.3	13.4±0.7	21.2±0.6	30.7±0.4	48.6±2.8	62.4±0.7	68.9±0.0
10	7.5	140	30	15.8±1.1	19.6±0.4	29.3±0.8	43.8±0.9	51.7±1.5	52.7±2.9	10.2±0.8	14.5±0.0	22.5±0.1	35.4±0.4	42.9±0.9	45.7±0.9
11	7.5	140	45	15.8±0.8	22.3±0.5	30.5±3.7	47.2±4.7	56.7±0.2	61.4±3.3	10.6±0.6	16.8±0.3	23.7±2.6	38.4±3.8	47.8±0.0	53.6±2.5
12	7.5	140	60	15.4±0.9	19.4±2.3	27.2±1.0	43.9±1.0	54.8±0.4	57.6±1.0	9.4±0.5	14.1±1.3	21.3±0.2	35.6±1.2	46.1±1.3	49.8±1.4
13	7.5	150	30	14.3±0.1	20.4±0.6	28.3±2.3	45.7±0.3	59.7±3.8	61.1±3.6	8.8±0.1	14.9±0.3	22.5±1.8	37.6±0.3	51.9±3.0	54.9±3.0
14	7.5	150	45	12.7±0.8	18.2±1.0	26.4±3.1	43.9±1.5	55.9±2.1	61.1±1.7	7.1±0.3	12.7±1.0	21.0±2.0	36.5±1.6	49.1±1.1	55.4±2.0
15	7.5	150	60	15.3±1.0	20.9±0.6	28.2±1.6	46.1±0.4	57.8±0.1	63.7±2.9	10.2±0.7	16.1±0.3	22.6±1.2	38.3±0.4	50.6±0.2	57.6±2.2
16	7.5	160	30	13.5±0.2	18.7±0.2	29.0±0.6	48.0±1.6	57.6±6.1	67.7±1.2	9.7±0.1	15.4±0.2	24.3±0.2	38.8±3.5	51.8±5.4	62.2±0.7
17	7.5	160	45	13.3±0.3	18.8±0.4	28.1±1.2	46.6±2.9	61.3±9.6	76.8±1.4	8.0±0.3	13.8±0.4	23.1±0.8	39.5±1.8	54.1±7.9	69.9±1.7
18	7.5	160	60	12.0±0.1	17.1±0.3	26.7±0.9	47.9±0.2	60.1±5.1	68.4±5.2	7.8±0.1	13.5±0.2	23.3±0.6	42.0±0.2	54.5±4.3	63.5±4.4
19	5	140	30	14.5±0.9	19.1±0.4	24.2±2.0	38.4±1.5	45.4±1.0	51.4±3.9	8.1±0.5	12.4±0.4	17.4±1.0	30.4±0.4	38.9±1.6	45.4±3.9
20	5	140	45	13.7±1.2	20.6±0.1	27.6±1.4	39.9±2.3	50.2±3.4	51.5±1.8	8.2±0.9	14.6±0.1	21.7±1.0	33.7±2.1	44.5±3.2	47.0±1.8
21	5	140	60	15.3±0.0	19.3±0.0	26.6±0.0	44.4±0.0	52.2±0.0	55.8±0.0	9.3±0.0	14.0±0.0	21.0±0.0	37.7±0.0	46.7±0.0	51.3±0.0
22	5	150	30	14.8±0.5	21.4±0.8	28.7±1.7	38.5±1.2	48.7±3.1	54.1±0.2	8.4±0.2	14.8±0.7	21.9±1.3	31.9±0.7	42.2±3.0	48.1±0.2
23	5	150	45	15.2±0.1	21.7±0.4	27.0±0.6	38.4±0.5	51.6±1.6	62.8±4.5	8.3±0.0	14.7±0.3	20.5±0.3	31.8±0.5	44.6±1.4	55.6±4.0
24	5	150	60	13.8±0.1	19.3±0.3	26.4±0.8	38.8±0.4	46.4±2.6	65.5±2.9	7.3±0.2	13.3±0.6	21.3±0.6	34.0±0.3	42.5±2.3	61.5±2.0
25	5	160	30	13.5±0.5	17.5±0.3	26.6±2.3	35.4±1.3	48.0±0.1	57.2±2.5	7.3±0.5	12.0±0.5	21.6±2.0	30.7±1.4	43.1±0.6	53.3±1.2
26	5	160	45	12.2±0.2	17.7±0.3	24.8±0.2	37.6±1.3	45.3±2.5	52.3±1.1	6.4±0.3	12.1±0.1	20.5±0.0	33.3±1.1	41.7±2.2	49.1±0.7

27	5	160	60	13.6±0.4	19.9±0.1	25.4±0.4	40.5±1.6	47.0±3.1	54.4±1.6	7.3±0.4	13.5±0.5	20.7±0.7	35.2±1.0	42.7±2.5	50.6±1.7
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Table 3. Overall yield of glucose and xylose (mol.%) obtained after HAS pretreatment and enzymatic hydrolysis.

Run	Pretreatment conditions			Overall yield of	
	S (wt.%)	T (°C)	t (min)	glucose mol.%	xylose mol.%
1	10.0	140	30	62.2±4.8	54.2±5.0
2	10.0	140	45	65.4±0.5	55.8±0.9
3	10.0	140	60	71.7±4.6	58.1±9.8
4	10.0	150	30	72.1±2.0	59.7±1.9
5	10.0	150	45	76.8±4.5	61.2±4.0
6	10.0	150	60	70.5±5.1	54.2±5.2
7	10.0	160	30	71.9±0.9	53.8±1.2
8	10.0	160	45	73.2±1.9	54.4±2.8
9	10.0	160	60	70.7±0.6	51.7±0.6
10	7.5	140	30	52.7±2.9	46.4±1.5
11	7.5	140	45	61.3±3.5	51.7±2.6
12	7.5	140	60	56.7±1.2	48.2±1.6
13	7.5	150	30	58.8±4.0	50.2±3.1
14	7.5	150	45	59.6±1.7	49.8±2.2
15	7.5	150	60	62.3±2.9	51.6±2.8
16	7.5	160	30	63.3±1.2	49.6±0.7
17	7.5	160	45	73.7±1.4	57.1±2.0
18	7.5	160	60	65.2±5.3	51.2±4.4
19	5.0	140	30	51.0±4.0	44.5±3.9
20	5.0	140	45	51.4±1.8	45.0±1.9
21	5.0	140	60	53.9±0.7	47.1±0.6
22	5.0	150	30	54.0±0.2	46.2±0.4
23	5.0	150	45	62.3±4.7	51.3±4.2
24	5.0	150	60	64.4±3.0	56.3±2.1
25	5.0	160	30	57.6±2.5	49.0±1.3
26	5.0	160	45	52.2±1.1	43.8±0.8
27	5.0	160	60	51.7±1.6	42.5±1.8

Table 4. The delignification (*Y1*) and fermentable sugar yield (*Y2*) as a function of HAS pretreatment conditions

Run	Pretreatment Conditions (independent variables)			Main responses	
	S (wt.%)	T (°C)	t (min)	Delignification (wt.%)	Overall sugar yield (g/100g) ^a
1	10.0	140	30	49.1±2.3	33.6±2.0
2	10.0	140	45	55.4±0.4	35.1±0.3
3	10.0	140	60	60.3±0.7	37.9±2.5
4	10.0	150	30	57.7±0.4	38.3±0.8
5	10.0	150	45	67.1±1.0	40.4±1.9
6	10.0	150	60	68.3±0.7	36.7±2.2
7	10.0	160	30	69.5±0.3	37.1±0.4
8	10.0	160	45	77.2±1.8	37.7±0.9
9	10.0	160	60	76.7±0.3	36.3±0.3
10	7.5	140	30	43.5±0.4	28.5±1.1
11	7.5	140	45	51.9±0.4	32.8±1.4
12	7.5	140	60	50.3±1.5	30.4±0.5
13	7.5	150	30	54.0±1.1	31.5±1.6
14	7.5	150	45	58.6±1.0	31.7±0.7
15	7.5	150	60	61.0±0.3	33.1±1.2
16	7.5	160	30	68.0±1.6	33.1±0.5
17	7.5	160	45	67.2±0.7	38.4±0.7
18	7.5	160	60	72.0±2.3	34.2±2.1
19	5.0	140	30	43.9±0.9	27.5±1.7
20	5.0	140	45	51.6±0.6	27.8±0.8
21	5.0	140	60	52.9±1.4	29.1±0.3
22	5.0	150	30	50.5±0.8	29.0±0.1
23	5.0	150	45	52.5±1.4	33.1±1.9
24	5.0	150	60	54.4±1.8	34.8±1.2
25	5.0	160	30	57.6±0.5	30.9±1.0
26	5.0	160	45	61.5±0.2	27.8±0.4
27	5.0	160	60	64.2±0.9	27.4±0.7

^a overall sugars yield calculated on the basis of 100 g dry native sugarcane bagasse considering the yield of glucan and xylan recovery in pretreatment and enzymatic hydrolysis yields.

Table 5. ANOVA of linear, quadratic and interaction effects on delignification (*Y1*) and overall sugar yield (*Y2*). SS - sums of squares; df - degrees of freedom; MS - mean squares.

	Factor	SS	df	MS	F	<i>p</i>
<i>Y1</i>	(1) S (L)	471.485	1	471.485	81.710	0.0000
	(2)T (L)	1334.654	1	1334.654	231.299	0.0000
	(3) t (L)	242.985	1	242.985	42.110	0.0000
	1L by 2L	46.281	1	46.281	8.021	0.0097
$R^2 = 0.94$	Error	126.945	22	5.770		
$R^2 \text{ adj} = 0.93$	Total SS	2222.351	26			
	Factor	SS	df	MS	F	<i>p</i>
<i>Y2</i>	(1) S (L)	239.968	1	239.968	56.114	0.0000
	(2)T (L)	23.041	1	23.041	5.388	0.0310
	T (Q)	18.538	1	18.538	4.335	0.0504
	2L by 3L	10.082	1	10.082	2.358	0.1403
	t (Q)	7.534	1	7.534	1.762	0.1994
	(3) t (L)	5.731	1	5.731	1.340	0.2607
$R^2 = 0.78$	Error	85.529	20	4.276		
$R^2 \text{ adj} = 0.72$	Total SS	390.423	26			

Table 6. ANOVA of predictor models for delignification (*Y1*) and fermentable sugar yield (*Y2*). SS - sums of squares; df - degrees of freedom; MS - mean squares; Fcalc - calculated F value; Ftab - F critical value.

	SV	SS	df	MS	Fcalc	Ftab	Fcalc/Ftab	R^2
<i>(Y1)</i>	Regression	2095.41	4	523.85	90.78	2.82	32	0.94
	Residual error	126.95	22	5.77				
	Total	2222.35	26					
	SV	SS	df	MS	Fcalc	Ftab	Fcalc/Ftab	R^2
<i>(Y2)</i>	Regression	304.89	6	50.82	11.87	2.60	4.6	0.78
	Residual error	85.53	20	4.28				
	Total	390.42	26					

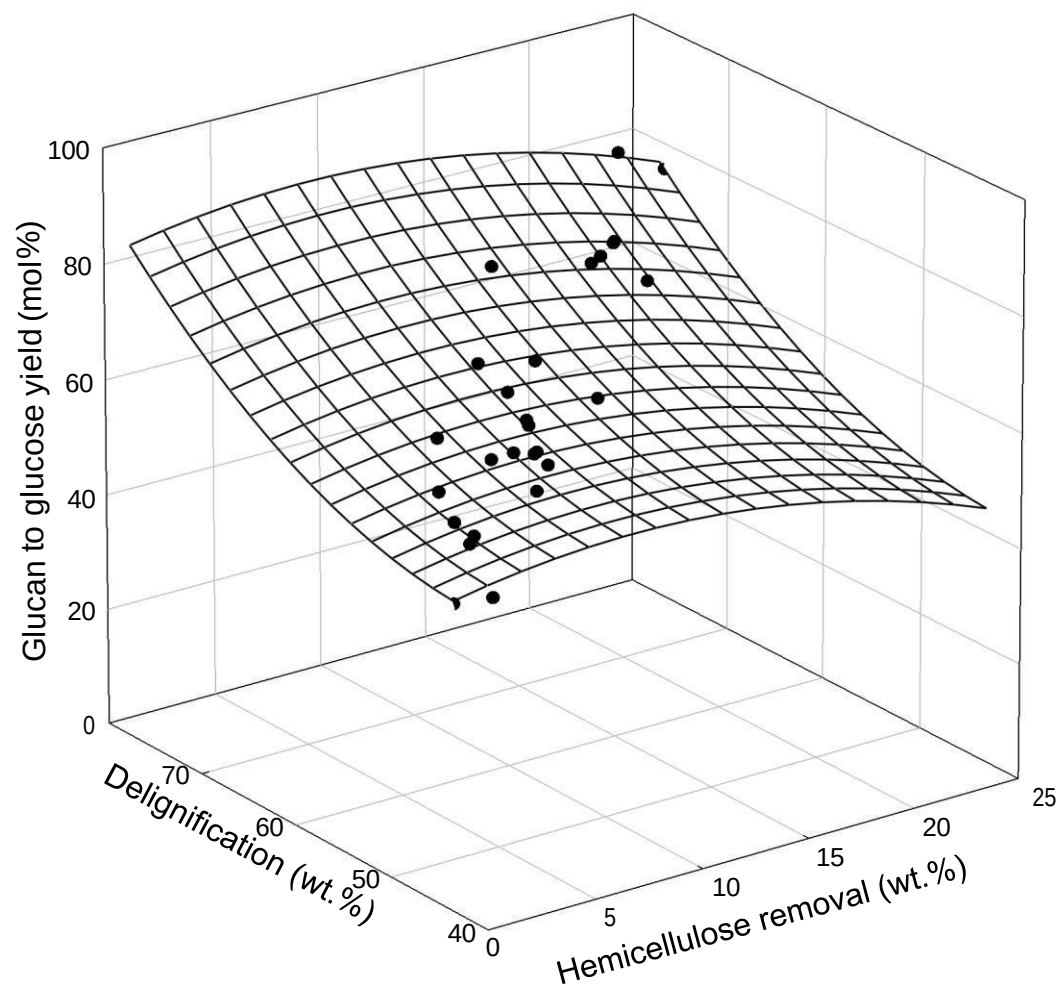


Figure 1. The graphical visualization of enzymatic hydrolysis yield in function of delignification and hemicellulose removal. The closed symbols represent the experimental points and surface is a mere representation of observed relation.