

† **Electronic supplementary information**

**In-situ Pretreatment during Distillation Improves Corn Fiber Conversion and
Ethanol Yield in Dry Mill Process**

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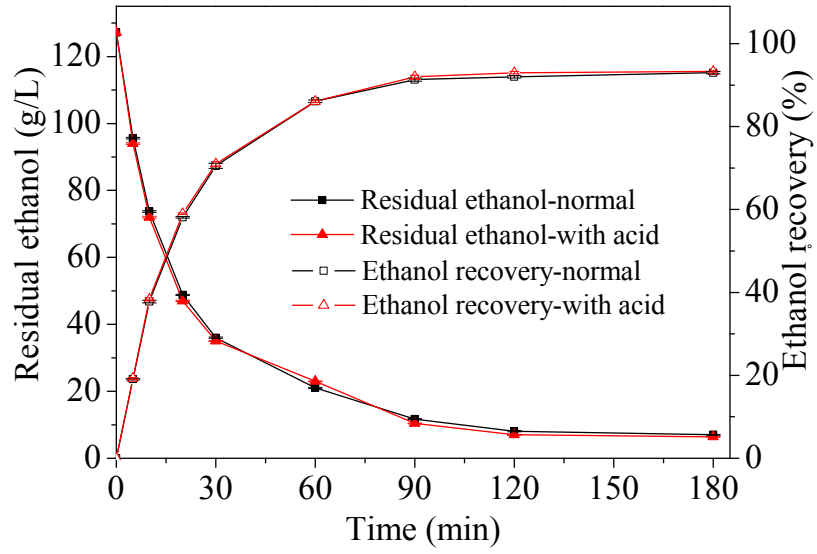
ESI Table 1† Effect of pretreatment conditions on composition of stillage.

Pretreatment conditions		Composition after NaOH pretreatment		Composition after H ₂ SO ₄ pretreatment	
T(°C)	NaOH or H ₂ SO ₄ loading, time	Glucan (%)	Xylan (%)	Glucan (%)	Xylan (%)
Control	--	14.39±0.07	11.66±0.09		
82	1.0%, 30 min	9.58±0.07	8.73±0.04	11.68±0.06	8.94±0.08
	1.0%, 60 min	9.85±0.05	7.80±0.04	11.46±0.11	8.68±0.07
	1.0%, 90 min	10.01±0.04	7.03±0.03	12.82±0.09	7.37±0.08
	1.5%, 30 min	8.02±0.05	6.86±0.05	10.84±0.04	7.82±0.06
	1.5%, 60 min	9.05±0.07	7.57±0.05	10.45±0.11	7.46±0.03
	1.5%, 90 min	8.83±0.08	6.74±0.04	10.93±0.06	7.78±0.05
	2.0%, 30 min	8.88±0.05	6.80±0.06	9.87±0.04	7.05±0.05
	2.0%, 60 min	8.25±0.03	6.85±0.06	11.03±0.07	7.37±0.04
	2.0%, 90 min	8.15±0.08	7.32±0.05	10.57±0.05	6.88±0.06
90	1.0%, 30 min	9.81±0.04	7.82±0.08	11.36±0.11	8.22±0.04
	1.0%, 60 min	10.47±0.06	8.51±0.07	11.19±0.15	7.97±0.06
	1.0%, 90 min	10.07±0.09	6.91±0.08	11.42±0.18	8.18±0.06
	1.5%, 30 min	9.24±0.05	6.81±0.06	11.12±0.11	7.88±0.03
	1.5%, 60 min	9.72±0.07	7.04±0.03	11.44±0.19	7.63±0.05
	1.5%, 90 min	9.36±0.09	7.65±0.04	11.23±0.09	7.21±0.05
	2.0%, 30 min	9.47±0.05	7.26±0.03	11.57±0.08	7.85±0.04
	2.0%, 60 min	8.78±0.18	6.76±0.05	10.12±0.08	6.87±0.04
2.0%, 90 min	8.91±0.09	6.97±0.05	11.42±0.12	7.26±0.03	
95	1.0%, 30 min	9.91±0.08	7.85±0.05	11.68±0.09	8.35±0.06
	1.0%, 60 min	9.85±0.05	7.76±0.05	10.45±0.06	7.35±0.03
	1.0%, 90 min	10.09±0.08	7.9±0.04	11.64±0.04	7.86±0.04
	1.5%, 30 min	9.42±0.07	7.56±0.06	10.9±0.05	7.34±0.04
	1.5%, 60 min	9.53±0.05	7.13±0.06	10.37±0.03	6.8±0.03
	1.5%, 90 min	9.42±0.04	7.53±0.05	10.3±0.05	6.74±0.05
	2.0%, 30 min	9.74±0.07	6.45±0.05	10.03±0.04	6.44±0.05
	2.0%, 60 min	9.53±0.05	6.63±0.04	8.95±0.04	5.8±0.04
2.0%, 90 min	8.88±0.07	7.16±0.06	10.31±0.07	6.12±0.06	
100	1.0%, 30 min	10.65±0.08	8.69±0.06	12.88±0.09	10.88±0.04
	1.0%, 60 min	10.12±0.05	8.39±0.05	11.06±0.12	8.18±0.04
	1.0%, 90 min	9.77±0.07	8.23±0.05	10.97±0.09	7.62±0.04
	1.5%, 30 min	9.69±0.08	8.17±0.04	11.87±0.11	9.1±0.06
	1.5%, 60 min	9.38±0.09	7.42±0.06	10.80±0.09	7.55±0.05
	1.5%, 90 min	9.11±0.08	8.04±0.06	10.09±0.06	6.82±0.06
	2.0%, 30 min	9.41±0.06	7.33±0.03	9.88±0.05	6.06±0.07
	2.0%, 60 min	8.88±0.07	6.98±0.05	9.65±0.06	5.52±0.09
	2.0%, 90 min	8.65±0.05	7.23±0.05	9.37±0.08	6.32±0.05

ESI Table 2† Effect of pretreatment conditions on enzymatic hydrolysis of stillage.

Pretreatment conditions		Enzymatic hydrolysis of NaOH pretreated stillage				Enzymatic hydrolysis of H ₂ SO ₄ pretreated stillage			
T(°C)	NaOH or H ₂ SO ₄ loading, time	Glucose (g/L)	Xylose (g/L)	Glucose yield (%)	Xylose yield (%)	Glucose (g/L)	Xylose (g/L)	Glucose yield (%)	Xylose yield (%)
Control	--	13.40±0.11	0.00±0.00	41.31±0.32	0.00±0.00				
82	1.0%, 30 min	14.09±0.11	0.79±0.03	83.88±0.46	4.50±0.05	15.81±0.15	1.51±0.04	61.33±0.22	8.40±0.02
	1.0%, 60 min	15.29±0.09	0.79±0.04	82.44±0.52	5.03±0.04	15.98±0.19	1.75±0.04	67.83±0.52	10.02±0.05
	1.0%, 90 min	16.59±0.11	1.02±0.05	84.32±0.45	7.21±0.05	17.23±0.20	2.79±0.10	68.43±0.36	18.83±0.18
	1.5%, 30 min	16.09±0.16	0.94±0.05	90.95±0.44	6.81±0.04	16.61±0.18	2.68±0.04	70.48±0.53	17.03±0.14
	1.5%, 60 min	16.13±0.18	0.97±0.04	90.55±0.51	6.37±0.05	16.26±0.14	2.83±0.06	69.00±0.62	18.51±0.16
	1.5%, 90 min	15.92±0.11	0.92±0.06	91.61±0.46	6.78±0.03	17.31±0.09	3.31±0.05	70.12±0.55	21.15±0.19
	2.0%, 30 min	15.86±0.07	0.86±0.05	90.74±0.42	6.29±0.04	15.14±0.16	2.84±0.04	77.98±0.52	20.02±0.21
	2.0%, 60 min	15.12±0.18	0.81±0.05	92.98±0.29	5.88±0.03	17.47±0.11	3.38±0.03	75.73±0.49	22.80±0.18
	2.0%, 90 min	15.62±0.14	0.87±0.04	97.38±0.33	5.91±0.03	17.81±0.09	4.20±0.04	76.65±0.59	30.43±0.22
90	1.0%, 30 min	17.54±0.14	0.94±0.06	90.91±0.43	5.97±0.03	17.62±0.18	2.55±0.04	78.86±0.35	15.42±0.09
	1.0%, 60 min	17.96±0.17	1.14±0.06	92.08±0.49	6.66±0.05	18.94±0.14	2.55±0.05	86.13±0.38	15.90±0.12
	1.0%, 90 min	17.68±0.11	1.03±0.03	89.21±0.51	7.41±0.05	19.13±0.16	3.59±0.05	85.79±0.41	20.60±0.18
	1.5%, 30 min	16.88±0.16	0.97±0.04	92.90±0.47	7.08±0.05	17.45±0.21	3.08±0.05	79.75±0.36	19.43±0.20
	1.5%, 60 min	16.72±0.14	0.83±0.04	87.43±0.42	5.86±0.06	19.03±0.15	3.81±0.04	84.50±0.42	24.82±0.19
	1.5%, 90 min	16.25±0.11	0.86±0.03	88.26±0.39	5.59±0.04	19.01±0.18	4.18±0.06	87.81±0.51	28.82±0.18
	2.0%, 30 min	16.84±0.19	0.71±0.05	90.38±0.43	4.86±0.03	18.77±0.18	3.72±0.06	82.50±0.37	23.56±0.21
	2.0%, 60 min	15.86±0.15	0.83±0.05	91.76±0.36	6.10±0.04	17.54±0.27	4.39±0.04	88.04±0.42	31.76±0.28
	2.0%, 90 min	16.15±0.12	0.81±0.05	92.17±0.44	5.78±0.05	18.71±0.18	4.88±0.04	83.26±0.44	33.41±0.31
95	1.0%, 30 min	19.37±0.11	1.17±0.06	79.71±0.53	5.95±0.04	19.81±0.14	4.25±0.06	69.20±0.41	19.93±0.13
	1.0%, 60 min	19.64±0.18	1.13±0.07	82.68±0.55	5.81±0.06	19.96±0.09	4.85±0.04	76.73±0.30	26.33±0.18
	1.0%, 90 min	18.68±0.16	0.64±0.09	75.56±0.46	3.23±0.05	22.39±0.14	6.06±0.06	78.67±0.35	37.19±0.39
	1.5%, 30 min	18.22±0.11	0.94±0.05	78.89±0.38	4.96±0.04	21.39±0.12	6.23±0.05	80.07±0.32	33.89±0.29
	1.5%, 60 min	18.01±0.09	1.03±0.04	77.09±0.36	5.76±0.04	19.99±0.11	6.19±0.04	78.66±0.22	36.30±0.24
	1.5%, 90 min	17.89±0.16	0.86±0.04	77.50±0.43	4.56±0.04	20.03±0.09	6.6±0.06	78.36±0.30	39.08±0.26
	2.0%, 30 min	16.91±0.11	0.97±0.04	70.81±0.54	6.00±0.05	19.92±0.09	6.98±0.05	80.99±0.54	43.25±0.38
	2.0%, 60 min	16.31±0.16	0.81±0.06	69.80±0.43	4.82±0.03	19.57±0.16	6.45±0.07	89.27±0.36	44.38±0.32
	2.0%, 90 min	17.43±0.18	0.86±0.06	80.10±0.55	4.79±0.06	21.92±0.18	6.44±0.04	86.73±0.34	41.99±0.41
100	1.0%, 30 min	20.22±0.11	1.24±0.06	77.45±0.38	5.69±0.04	21.85±0.27	3.91±0.06	69.24±0.36	14.29±0.11
	1.0%, 60 min	19.09±0.16	0.49±0.04	76.95±0.28	2.33±0.03	22.61±0.18	5.52±0.04	83.45±0.42	26.92±0.18
	1.0%, 90 min	19.39±0.18	0.76±0.04	81.01±0.22	3.69±0.02	23.58±0.17	7.16±0.04	87.72±0.52	37.47±0.24
	1.5%, 30 min	18.45±0.11	0.80±0.06	77.66±0.28	3.91±0.03	22.68±0.19	5.93±0.05	77.98±0.39	26.01±0.21
	1.5%, 60 min	18.65±0.15	0.94±0.07	81.10±0.36	5.06±0.04	22.79±0.14	7.74±0.03	86.11±0.44	40.93±0.38
	1.5%, 90 min	18.75±0.14	0.75±0.09	83.92±0.3	3.72±0.03	22.66±0.11	8.12±0.04	91.62±0.49	47.68±0.41
	2.0%, 30 min	17.37±0.16	1.02±0.05	75.30±0.42	5.55±0.04	21.87±0.14	7.89±0.04	90.34±0.53	51.93±0.48
	2.0%, 60 min	16.51±0.11	0.74±0.05	75.88±0.47	4.23±0.05	22.21±0.18	8.12±0.06	93.89±0.56	58.74±0.51
	2.0%, 90 min	17.66±0.15	0.88±0.05	83.36±0.51	4.86±0.05	22.63±0.15	8.22±0.03	96.57±0.46	51.91±0.47

Enzymatic hydrolysis of whole stillage was performed at 15% solid loading for 72 h. Cellulase and xylanase dosages were both 20 mg protein/g glucan.



ESI Fig. 1† Effect of acid addition (1%, w/w) on corn-based ethanol distillation. **With the addition of 1% (w/w) H_2SO_4 , the fermentation broth obtained from SSF with 30% (w/w) solid loading of corn was distilled at 95 °C under atmospheric pressure.**

ESI Table 3† Dynamic change of acid concentration during ethanol distillation.

Distillation time (min)	Corn dry weight (w/w)	H ₂ SO ₄ (%)		
0	10.8	0.6	0.8	1.0
5	11.5	0.6	0.9	1.1
10	12.0	0.7	0.9	1.1
20	12.8	0.7	1.0	1.2
30	13.3	0.7	1.0	1.2
60	13.9	0.8	1.0	1.3
90	14.3	0.8	1.1	1.3