Electronic Supplementary Information

Enhanced production and separation of short-chain glucan oligomers from corn stover in unacidified LiBr molten salt hydrate via pre-extraction of hemicellulose

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1. Method:

1.1 Component analysis of biomass

The components in corn stover or pretreated corn stover were quantified following the method used in our previous work.[1] The sample was firstly dried in an oven at 80 °C for 12 h to remove adsorbed water. Then 0.3 g of dried sample was added into a 100 mL glass beaker with 2 mL of 72 wt.% H_2SO_4 before being transferred into a preheated water-bath at 30 °C for 2 h. The mixture was hand-stirring with a glass bar every 15 min to ensure a sufficient hydrolysis. After concentrated H_2SO_4 hydrolysis, the mixture was washed with 56 g of deionized water to dilute the acid concentration to 4 wt.%, followed by being transferred into a 120 mL thick-wall glass reactor (Synthware Co., Ltd., China). The reactor was put into a preheated oil bath at 121 °C for 1 h with magnetic stirring at 300 rpm. After hydrolysis, the hydrolysate was separated from solid part with filtration. Sugars dissolved in hydrolysate was quantified by HPLC. Cellulose and hemicellulose contents were calculated based on monosaccharides amount as follows:

$$Cellulose\ content(\%) = \frac{W_{glucose} \times 0.9}{W_{total}} \times 100\%$$

$$Hemicellulose\ content(\%) = \frac{W_{xylose} \times 0.88}{W_{total}} \times 100\%$$

Here $W_{glucose}$ and W_{xylose} refer to masses of glucose and xylose quantified by HPLC and W_{total} refers to the mass of dried sample before hydrolysis.

Besides, the solid part obtained after hydrolysis was washed dried and weighted. Then the solid was put into a muffle furnace at 575 °C for 4 h and weighted. The lignin content was calculated as:

$$Lignin\ content(\%) = \frac{W_{before} - W_{after}}{W_{total}} \times 100\%$$

Here, W_{before} and W_{after} refer to the weight of the solid residue before and after calcination. W_{total} refers to the mass of dried sample before hydrolysis.

1.2 Quantification of glucan oligomer DP

As shown in **Figure S1**, during phenyl isocyanate functionalization, the hydroxyl functional groups in glucan oligomer were reacted with the carbonyl groups in phenyl isocyanate through an addition reaction. The molecular weight of functionalized oligomer can be calculated as $162n+18+(3n+2)\times119 = 519n+256$.
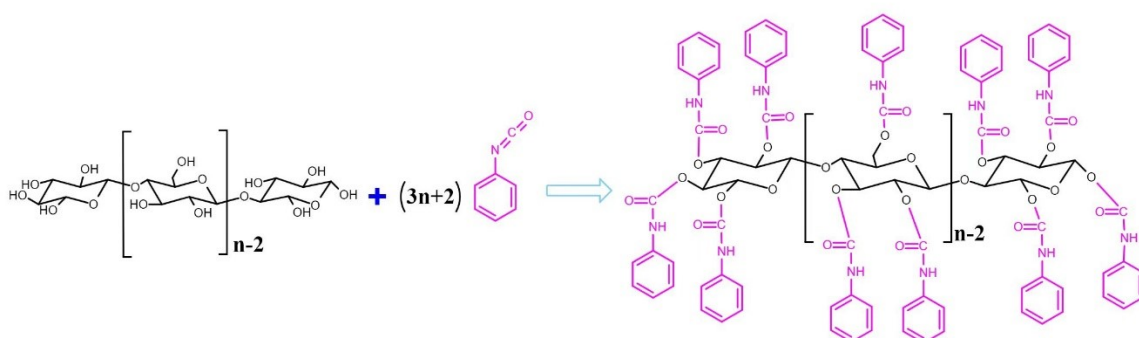
Figures:

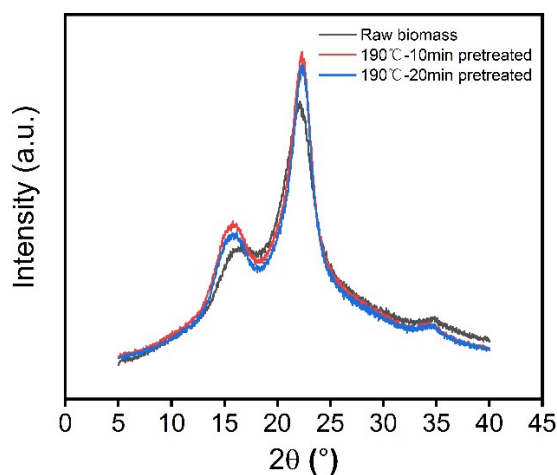**Figure S1**. Scheme of glucan oligomer functionalization with phenyl isocyanate

Figure S2. The XRD pattern of raw 190 ℃-10 min and 190 ℃-10 min pre-extracted corn stover.

Tables:

Table S1 The effect of substrate addition on glucan oligomer yield [a]

Mass ratio	Raw corn stover	Pre-extracted corn stover
60:1	81.6%	90.1%
30:1	76.2%	88.0%
15:1	67.6%	88.1%
10:1	--	75.9%

[a] The mass ratio refers to the mass of MSH to the mass of cellulose dosage in substrate.

1.   W. Li, Q. Liu, Q. Ma, T. Zhang, L. Ma, H. Jameel and H.-m. Chang, *Bioresource technology*, 2016, **219**, 753-756.