Coproduction of furfural, phenolated ligninand and fermentable sugars from bamboo with one-pot fractionation using phenol-acidic 1,4-dioxane

Li Ji^{a, b}, Pengfei Li^{b, c}, Fuhou Lei^c, Xianliang Song^b, Jianxin Jiang^{a, b*}, Kun Wang^{b*}
^aBeijing Advanced Innovation Center for Tree Breeding by Molecular Design, Beijing Forestry University, Beijing 100083, China

^bDepartment of Chemistry and Chemical Engineering, MOE Engineering Research Center of Forestry Biomass Materials and Bioenergy, Beijing Forestry University, Beijing 100083, China

^cGuangxi Key Laboratory of Chemistry and Engineering of Forest Products, College of Chemistry and Chemical Engineering, Guangxi University for Nationalities, Nanning 530006, China

*Corresponding author

 $\label{linear_complex} \mbox{Jianxin Jiang}^*, e\mbox{-mail: jiangjx} 2004 @ hotmail.com$

Kun Wang*, e-mail: wangkun@bjfu.edu.cn

Method of Enzyme Activity Analysis	2
Reaction Conditions During Fractionation	3
Material Composition Analyses	
GPC Analyses	5
Enzyme Activity Analyses	6

Enzyme Activity Analyses

The release of p-nitrophenol from p-nitrophenyl- β -D-galactopyranoside (pNPG) was monitored to determine the β -glucosidase activity. The enzyme solution of 0.5 mL was incubated with 1 mL pNPG (1 mmol·L⁻¹) of sodium acetate, pH 4.8, and 1.5 mL H₂O at 50 °C for 10 min. The 2 mL of sodium carbonate (1 mol·L⁻¹) was added to stop the enzyme reaction, and the colour developed at 400 nm. The exo- β -1,4-glucanase activity was assayed using p-nitrophenol-D-cellobioside (pNPC) as substrate to determine its ability to hydrolyze cellobiose. The enzyme solution (0.5 mL) was incubated with 4.5 mL pNPC (1 mmol·L⁻¹) of sodium acetate, pH 4.8, at 50 °C for 30 min. The 5 mL of sodium carbonate (1 mol·L⁻¹) was added to stop the enzyme reaction, and the colour developed at 400 nm.

Table S1. The specific reaction condition during fractionation process.

Without phenol addition					With phenol addition				
Entry	1,4-dioxane solvent addition/mL	T/°C	Time/h	Severity factor	Phenol addition (%, w/v)	T/°C	Time/h	1,4-dioxane solvent addition/mL	Severity factor
1	900	80	5	1.89	1.0	80	2	900	1.49
2	700	80	5	1.89	2.0	80	2	900	1.49
3	500	80	5	1.89	2.5	80	2	900	1.49
4	500	120	3	2.84	1.0	100	2	900	2.08
5	900	80	3	1.67	15	100	2	900	2.08
6	500	120	1	2.37	2.5	100	2	900	2.08
7					1.0	120	2	900	2.67
8		-			1.5	120	2	900	2.67

9 1.0 140 2 900 3.26

The material composition analysis was used to determine the effect of fractionation conditions on chemical component of substrates. The yields of residual lignin, dissolved lignin and glucan were detected according to NREL method.

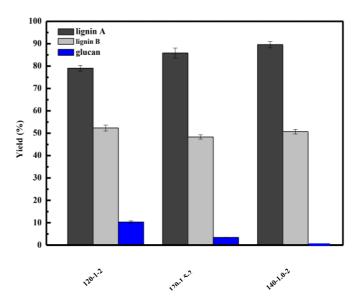


Figure S1. The effects of reaction temperature (120 °C and 140 °C) and phenol additions (1.0% and 1.5%, w/v) on lignin and glucan yield of moso bamboo. lignin A: residual lignin; lignin B: dissolved lignin (phenolated lignin included).

The GPC analysis was used to verity the effect of phenol addition on molecular weight of fractionated lignin. The GPC chromatogram of lignin I and lignin II were recorded on Angilent HPLC technique on a PLgel-Mixed-D column.

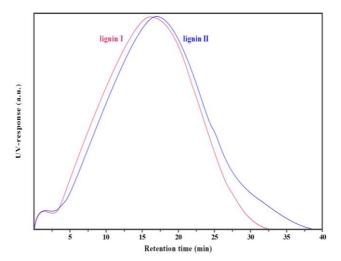


Figure S2. Molecular weight distributions for lignin fractions lignin I and lignin II.

The enzyme activity analysis was used to verity the effect of fractionation conditions on conversion of carbohydrate pulps to fermentable sugars. The β -glucosidase activity, the exo- β -1,4-glucanase activity were recorded on UV spectrophotometer.

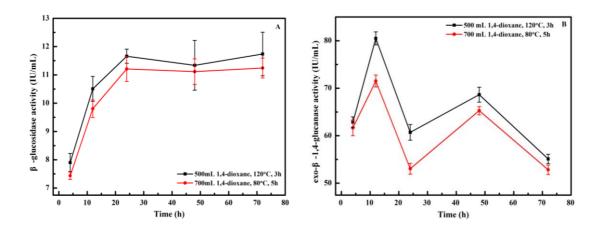


Figure S3. The effects of enzyme activities on moso bamboo hydrolysis. A: The β -glucosidase activity during the time period 0 to 72 h of enzymatic hydrolysis. B: The exo- β -1,4-glucanase activity during the time period 0 to 72 h.