# **Response Surface Optimization of Alkali Extraction and Characterization of Poplar Hemicellulose**

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Hemicellulose was isolated from poplar powder using alkaline hydrolysis coupled with alcohol precipitation. Response surface methodology was applied to study the effects of sodium hydroxide concentration, reaction time, and temperature on the extraction yield. The optimal conditions for the extraction of the hemicellulose from poplar powder were an alkaline mass fraction of 9.5%, reaction time of 4 h and 12 min, and temperature of 78 °C. The extraction yield reached 52.8% under this optimal condition. Fourier-transform infrared spectroscopy, nuclear magnetic resonance, thermogravimetric analysis, and sugar component analyses showed that the obtained hemicellulose with excellent water absorption and heat resistance consisted mainly of 4-*O*-methyl-glucurono-xylan, and the molar ratio of xylose to glucuronic acid on the molecule chains was 3.95.

Keywords: Poplar; Hemicellulose; Alkaline extraction; Response surface methodology

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#### INTRODUCTION

With the exhaustion of petrochemical resources, the serious pollution of environment, and the looming global energy crisis, the development of renewable biomass resources for fuels and materials harvesting is imperative (Hansen and David 2008; Farhat *et al.* 2017; Ibn Yaich *et al.* 2017). Efficient exploitation and utilization of materials and energies from renewable resources are of great importance for the sustainable development of human society (Tunc *et al.* 2010; Cherubini 2010; Putro *et al.* 2016). Cellulose, hemicellulose, and lignin are the main components of plant cell walls, making them the most abundant biomass resources in nature. Hence, the utilization of cellulose, hemicellulose, and lignin in papermaking, food packaging, and bio-medical fields has become a research hotspot in recent years.

Different from cellulose and lignin, hemicellulose is made of homogeneous or heterogeneous polysaccharides connected by different monosaccharides with diverse binding methods. The typical structural unit of hemicellulose includes neutral sugars (*D*-xylose, *L*-arabinose, *D*-galactose) and glucuronic acid (Scheller and Ulvskov 2010). Its characteristics of non-toxicity and biodegradability make hemicellulose suitable for food functional packaging materials, edible film, and biological medicine (Mikkonen and Tenkanen 2012). However, the diverse compositions and amorphous structures of hemicellulose increase the difficulty of its separation, purification, and modification. Therefore, research on hemicellulose is less adequate than that on cellulose and lignin. The current extraction methods of hemicellulose include steam explosion (Wang *et al.* 2010; Martin-Sampedro *et al.* 2014), hot water treatment (Liu 2010; Liu *et al.* 2012; Krogell *et al.* 2013; Cheng *et al.* 2014; Azhar *et al.* 2015), ultrasonic treatment (Pei *et al.* 2015), alkaline extraction (Methacanon *et al.* 2003; Krawczyk *et al.* 2008; Cheng *et al.* 2011;

Johakimu and Andrew 2013; Sun *et al.* 2013), acid extraction (Walton *et al.* 2010; Guerra-Rodríguez *et al.* 2012; Wang *et al.* 2012), and organic solvent extraction (Saake *et al.* 2001; Hu *et al.* 2009; Xu *et al.* 2013). Of all the aforementioned approaches, alkaline extraction has the advantage of higher extraction yields, less destructiveness of molecular structures, and a higher polymerization degree of the obtained hemicellulose (Sun *et al.* 2016).

Poplar is a hardwood of the genus *Populus*, which is mainly distributed in the north temperate zone. Due to its fast-growing nature and good adaptability, poplar is widely planted in China as an important artificial forest species. During the extensive application of wood processing, a large amount of poplar powder is produced and discarded, resulting in the waste of biomass resources and environmental pollution. The hemicellulose content in discarded poplar powder is generally 20% to 25% and sometimes up to 35% (Alekhina *et al.* 2014). The effective extraction of hemicellulose from these wastes is of great significance to the full utilization of poplar powder. Alkaline extraction of hemicellulose from poplar was reported (Sun *et al.* 2001), in which poplar hemicellulose was extracted with 8.5% mass fraction of NaOH solution at 20 °C. However, the influence of alkaline pretreatment on the extraction efficiency of hemicellulose from poplar was not thoroughly studied, and a systematic characterization analysis of the obtained hemicellulose has not been reported.

Response surface methodology (RSM) is a combination method of mathematical and statistical theories to solve multivariable problems (Thompson 1982). Using reasonable experimental design and processing of experimental data, the functional relationship between factors and response values is fitted by multiple quadratic regression equations (Thompson 1982). The optimal process parameters are determined through the analysis of regression equations. Due to fewer test runs, shorter duration, and higher precision, RSM is used to reduce experimental cost, optimize processing conditions, improve product quality, and solve practical problems in food and chemical manufacturing (Mu 2001). In the development and utilization of biomass resources, RSM has been successfully applied to enhance the yield of hemicellulose extracted from *Cornus officinalis* residue (Shao *et al.* 2011), to improve dilute acid pretreatment of straw hemicellulose (Won *et al.* 2012), and to optimize NaOH extraction of bagasse hemicellulose (Yao *et al.* 2015).

In this study, hemicellulose was extracted from poplar powder by alkaline hydrolysis followed by alcohol precipitation. The parameters of alkali concentration, reaction time, and temperature in the extraction process were optimized by RSM. The composition and structure of the obtained hemicellulose were analyzed.

#### **EXPERIMENTAL**

#### **Materials and Instruments**

Poplar powder was obtained from five-year-old poplar in the Hebei region, the hemicellulose content of which was 30.5%. The particle size was in the range 0.2 mm to 0.8 mm. The NaOH, HCl, NaClO, H<sub>2</sub>SO<sub>4</sub>, Ba(OH)<sub>2</sub>, and KH<sub>2</sub>PO<sub>4</sub> were purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China), and glacial acetic acid was purchased from Beijing Chemical Plant (Beijing, China). The 95% ethanol was purchased from Tianjin Oke Chemical Reagent Co., Ltd. (Tianjin, China); standardized reagents for chromatographic analysis were purchased from Sigma Co., Ltd. (Beijing, China). The instruments used in this study are listed in Table 1.

Name	Model	Manufacturer
High Speed Refrigerated Centrifuge	CR 22G	Beijing Tianlin Hengtai Technology Co., Ltd. (Beijing, China)
Fourier Transform Infrared Spectroscope	iN10 MX	Thermo Scientific (Waltham, MA, USA)
Thermogravimetric Analyzer	Q50	TA Instruments (Newcastle, DE, USA)
Nuclear Magnetic Resonance Spectrometer	AV 600	Bruker Instruments (Karlsruhe, Germany)
Liquid Chromatograph	Agilent 1200	Agilent Technologies (Santa Clara, CA, USA)

#### **Table 1.** Instruments Used for the Experiments and Analysis

#### **Extraction of Poplar Hemicellulose**

Hemicellulose was obtained from poplar powder through an alkaline hydrolysis followed by the alcohol precipitation approach (Fig. 1). The poplar powder was Soxhlet extracted with toluene and ethanol (2:1, v/v) for 6 h. The resulting defatted powder was dried for 12 h at 60 °C and then re-extracted with 0.6% (w/v) NaClO solution for 1 h at 75 °C. During the procedure, the solid-liquid ratio was 1:20 (w/v), and the pH was adjusted to 4.0 with acetic acid. After being filtered, rinsed, and dried, the filter residue was put into NaOH solution for a certain period of time at a certain temperature. The solid-liquid ratio was also 1:20 (w/v). Until the end of the reaction, the excess alkali was neutralized with HCl, and the pH was adjusted to 5.5. After refiltration, the resultant filtrate (containing hemicellulose) was precipitated with 95% (v/v) ethanol (1:3, v/v). The mixture was left to stand for 12 h before polar hemicellulose was obtained by centrifugation followed by drying the filter residue.





#### **Response Surface Experimental Design**

Results of an earlier single-factor experiment showed that the hemicellulose extraction yield reached the peak under the conditions of 9% NaOH with a reaction time of 4 h and 70 °C, respectively (Hu 2017). According to the Box-Behnken central

combinatorial experiment design principle (Box and Behnken 1960; Ferreira *et al.* 2007), NaOH concentration (A), reaction time (B), and temperature (C) were selected as three independent variables of the three-level factorial design model in RSM, and the hemicellulose extraction yield (%,  $R_x$ ) was the response value. Each experimental condition was done in three parallel tests, and the average value was reported. Table 2 presents the actual levels, corresponding to the codes of the process variables.

	Factors						
A: NaOH Concentration (%)	B: Reaction Time (h)	C: Temperature (°C)					
-1	7	3	60				
0	9	4	70				
1	11	5	80				

The response value  $R_x$  was calculated as Eq. 1,

$$R_{\rm x} = \frac{m_{\rm x}}{m_0} \times 100\% \tag{1}$$

where  $R_x$  is the extraction yield of hemicellulose (%),  $m_x$  is the total mass of xylose, mannose, arabinose, and glucuronic acid of extracted hemicellulose (g), and  $m_0$  is the total mass of xylose, mannose, arabinose, and glucuronic acid of defatted poplar powder (g).

The response surface quadratic model was analyzed by using Design-Expert software (State-Ease, New York, NY).

#### **Analytical Methods**

#### Gel permeation chromatography (GPC)

The molecular weight of hemicellulose was determined by GPC with a TSKG-5000 PWxL gel column (Beijing, China) under a pressure of 518 psi at 35 °C. The flow phase was 0.02 mol/L KH<sub>2</sub>PO<sub>4</sub> aqueous solution at a pH of 6.0, with velocity of 0.6 mL /min. The injection volume was 20  $\mu$ L.

#### Ion chromatography (IC)

The chemical composition of hemicellulose was analyzed by IC, and sugars were released from samples by acid hydrolysis. Hemicellulose of 300.0 mg  $\pm$  10.0 mg was solubilized in 84 g  $\pm$  0.04 g of distilled water and 3.00 mL  $\pm$  0.01 mL of H<sub>2</sub>SO<sub>4</sub> (72%, w/v). After being mixed, the sample was placed in the autoclave for 1 h at 121 °C. Then the pH of the reaction product was adjusted from 5 to 6 with Ba(OH)<sub>2</sub>. After centrifugation, the supernatant was filtered by a microporous membrane with a pore size of 0.22 µm. The content of dextran and xylose in hemicellulose was calculated by the filtrate and the determination of glucuronic acid is available in the literature (Li *et al.* 2007).

Aminex HPX-87P column (300 mm  $\times$  78 mm) (Bio Rad Laboratories, Hercules, CA, USA) was maintained at 80 °C. Degassed ultrapure water was used as the flow phase with a flow rate of 0.4 mL/min and injection volume of 0.5 µL. The assorted monitor was a differential refraction detector. The standard elution time was calibrated with *L*-arabinose, *D*-glucose, *D*-xylose, *D*-galactose, and *D*-mannose. The type of monosaccharide in the sample was determined by comparing the retention time of the standard substance and that of the sample; the percentage of monosaccharide in the sample was calculated according to the peak area.

#### Fourier-transform infrared spectroscopy (FT-IR)

The chemical functional groups of hemicellulose were analyzed by FT-IR. The spectra were obtained at a resolution of 4 cm<sup>-1</sup> with 32 scans in the range from 4000 cm<sup>-1</sup> to  $450 \text{ cm}^{-1}$ .

#### *Nuclear magnetic resonance (<sup>1</sup>H-NMR)*

Hemicellulose amounts of 10 mg were placed in the 5 mm diameter NMR tube. The solid sample was dissolved in 1 mL  $D_2O$  for analysis.

#### Thermogravimetric analysis (TGA)

Thermal stability of the hemicellulose was carried out on a thermogravimetric analyzer. The test temperature ranged from 40 °C to 600 °C with the nitrogen flow rate maintained at 100 mL/min and the heating rate of 20 °C/min.

#### **RESULTS AND DISCUSSION**

#### **Response Surface Optimization of Extraction Conditions**

Response surface results and variance analysis

In this paper, a Box-Behnken experimental design was used to optimize the NaOH concentration (A), reaction time (B), and temperature (C) in the extraction of hemicellulose from poplar powder by evaluating the extraction yield (%)  $R_x$  of hemicellulose. The statistical treatment combinations of the test variables along with the measured response values, expressed as the extraction yield of each combination, are summarized in Table 3.

	A. NaOH Concentration	B. Reaction Time	C: Temperature	Rx: Extraction Yield
No.	(%)	(h)	(°C)	of Hemicellulose (%)
1	9.0	3.0	60.0	35.7
2	9.0	5.0	80.0	45.9
3	9.0	5.0	60.0	35.4
4	9.0	4.0	70.0	51.9
5	7.0	3.0	70.0	16.9
6	7.0	4.0	80.0	30.6
7	7.0	4.0	60.0	34.5
8	7.0	5.0	70.0	28.7
9	11.0	3.0	70.0	29.8
10	11.0	4.0	60.0	37.1
11	9.0	4.0	70.0	53.5
12	11.0	5.0	70.0	32.4
13	9.0	4.0	70.0	48.6
14	11.0	4.0	80.0	45.0
15	9.0	4.0	70.0	51.5
16	9.0	3.0	80.0	34.8
17	9.0	4.0	70.0	52.1

The application of RSM produced the following regression equation (Eq. 2), which was an empirical relationship between extraction yield and the test variables in coded units,

 $R_{\rm x} = -399.47 + 54.254A + 87.23B + 0.3065C - 1.15AB + 0.145AC$ 

$$+ 0.285BC - 3.215A^2 - 11.71B^2 - 0.0186C^2$$

(2)

where  $R_x$  is the predicted poplar hemicellulose extraction yield, and *A*, *B*, and *C* are the coded values for the three variables, *i.e.*, NaOH concentration (%), reaction time (h), and temperature (°C) respectively.

The analysis of variance (ANOVA) data are shown in Table 4.

Source	Sum of Square	DF	Mean Square	<i>F</i> -value	P-value				
Model	1720.30	9	191.14	72.37	< 0.0001**				
A	141.12	1	141.12	53.43	0.0002**				
В	79.38	1	79.38	30.06	0.0009**				
С	23.12	1	23.12	8.75	0.0211*				
AB	21.16	1	21.16	8.01	0.0254*				
AC	34.81	1	34.81	13.18	0.0084**				
BC	32.49	1	32.49	12.30	0.0099**				
A <sup>2</sup>	696.34	1	696.34	263.65	< 0.0001**				
B <sup>2</sup>	577.36	1	577.36	218.60	< 0.0001**				
C <sup>2</sup>	14.57	1	14.57	5.52	0.0512				
Residual	18.49	7	2.64	-	-				
Lack of fit	5.56	3	1.85	0.57	0.6621				
Pure error	12.93	4	3.23	-	-				
Cor total	1738.78	16	-	-	-				
*Means significant (P < 0.05), **means highly significant ( $P < 0.01$ )									

**Table 4.** Variance Analysis of the Response Surface Model

The statistical significance of each variable in the model to the response value was verified by the F test (Qi *et al.* 2009). The larger F-value and the smaller P-value indicated that the significance of the influence of the variable on the response value was higher (Majumder and Goyal 2008). The F-value of the model was found to be 72.47 and the P-value was less than 0.0001, which indicated that the model terms were highly significant. The coefficient of the variation (CV) indicated the degree of precision with which the treatments were compared. A relatively lower value of CV, 4.16%, indicated a better precision and reliability of the experiments (Hou and Chen 2008).

The good fitting of the models was checked by the coefficient of determination  $(R^2)$ . The  $R^2$  is always between 0 and 1, and the closer the  $R^2$  value is to 1.0, the better the model predicted the response. Normally, a regression model with an  $R^2$  higher than 90% is regarded as a high correlation. The  $R^2$  value of the model was 98.94%, which implied that only 1.06% of the total variation could not be attributed to the model, and the model fit well to the observed data.

The lack of fit was non-significant (P-value = 0.6621 > 0.05), which illustrated that the RSM model was reliable and could be used to predict the actual situation of extracting hemicellulose from poplar powder (Yao *et al.* 2015).

As shown in Table 4, the influence of selected factors for hemicellulose extraction yield followed the order of *A* (NaOH concentration) > *B* (reaction time) > *C* (temperature) according to the F-value. In the present work, factors *A*, *B*, *AC*, *BC*,  $A^2$ , and  $B^2$  (*P*-value < 0.01) had highly significant impacts on extraction and factors *C* and *AB* (*P*-value < 0.05) had significant influence on the extraction yield.

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#### Interactions between variables

The regression model was analyzed by the software, Design-Expert, and the RSM three-dimensional (3D) graphs were obtained (Figs. 2, 3, and 4). Various response surface plots and contour plots showed the extraction yield plotted as functions of interactive variables. All three response surface diagrams have extreme points (the highest point of response surface, as well as the center point of the smallest ellipse of contour line). The extreme point demonstrated that the maximum of productive hemicellulose would exist above the selected condition.

As shown in Fig. 2, under the relatively short reaction time, alkaline concentration had little effect on the extraction yield of hemicellulose, and the increase in alkaline concentration could not improve the extraction yield effectively. However, when the reaction time was up to 4 h, higher NaOH concentrations enhanced the extraction yield, suggesting that the dissolution of hemicellulose in alkaline solution required plenty of time. Moreover, excessive reaction time led to reduced extraction yield, probably due to the loss of acetyl and other groups on hemicellulose during the process.



Fig. 2. Contour map and 3D diagram of the relationship between NaOH concentration and reaction time



Fig. 3. Contour map and 3D diagram of relationship between NaOH conc. and temperature



Fig. 4. The contour map and 3D diagram of the relationship between reaction time and temperature

Figure 3 shows that when the temperature was kept constant, the alkaline concentration had a significant influence on the extraction yield, and the optimal NaOH concentration was around 10%. At a certain concentration of NaOH, the extraction yield increased to a peak value and then decreased with the further increases in temperature. However, the elliptical curvature of contour line shown in Fig. 4 suggested that reaction time was more influential than the temperature with respect to the extraction yield.

In summary, the effect of NaOH concentration on the extraction yield of hemicellulose was the most important with the sharp response surface curve observed. While the reaction time and the temperature took the second and third places with their gentle response surface curves. The above inference revealed from graphs was consistent with the variance analysis of the response surface model.

#### Optimization and verification of extraction conditions

Through the calculation using the RSM analysis, the optimal conditions of poplar hemicellulose extraction were found to be a NaOH concentration of 9.47%, reaction time of 4.21 h, and 78 °C. The predicted extraction yield was 53.0% in this case. The reliability and practicality of the RSM model required validation. Adopting the predicted conditions and considering the convenience of practical operation, the authors amended the optimal experimental conditions, *i.e.* NaOH concentration of 9.5%, reaction time of 4 h 12 min, and 78 °C. The average extraction yield of three experiments was 52.8%, which was notably higher than that in the existing literature (Duan *et al.* 2013; Zhang 2016). The experimental value was only 0.23% different from the theoretical value, which demonstrated that the model was effective and reasonable.

#### **Characterization of Poplar Hemicellulose**

All the hemicellulose samples used for characterization were extracted from poplar powder under the amended optimal experimental conditions (*i.e.*, NaOH concentration of 9.5%, reaction time of 4 h 12 min and 78 °C).

#### Molecular weight distribution of hemicellulose

The molecular weight distribution of hemicellulose was evaluated by gel permeation chromatography (GPC). The measured weight-average molecular weight ( $M_w$ ) was 23,380 and the number-average molecular weight ( $M_n$ ) was 7560 Daltons. This illustrated that the alkaline hydrolysis and alcohol precipitation method was less destructive to the structure of hemicellulose. A wide dispersion coefficient, 3.09, was consistent with a previous report of alkaline extracted hemicellulose (Peng 2010).

#### Compositional analysis of hemicellulose

The monosaccharide composition of hemicellulose was analyzed by ion chromatography (IC), and the results are summarized in Table 5. Xylose was the main structural unit composed of hemicellulose, which accounted for 77.4%. Glucuronic acid is mainly 4-*O*-methyl glucuronic acid and accounted for 19.6%. Other monosaccharides such as glucose, rhamnose, and arabinose were less than 1.0%, and only galactose was more than 1.0%. The IC analysis indicated that the main composition of extracted poplar hemicellulose was 4-*O*-methyl-glucurono-xylan. The molar ratio of xylose to glucuronic acid on the molecule chains was found to be 3.95. The branching degree of extracted hemicellulose was not high, which was beneficial to further modification due to the lower steric hindrance (Wu 2014).

## **Table 5.** Content of Neutral Sugar and Glucuronic Acid in Extracted Hemicellulose

Xylose (%)	Glucuronic Acid (%)	The Molar Ratio of Xylose to Glucuronic Acid	Glucose (%)	Galactose (%)	Arabinose (%)	Rhamnose (%)
77.38	19.60	3.95	0.30	1.41	0.74	0.57

#### Chemical structural of hemicellulose

The FT-IR spectrum of the extracted poplar hemicellulose is shown in Fig. 5.



Fig. 5. The FT-IR spectrum of the extracted poplar hemicellulose

The absorption peak at 3425 cm<sup>-1</sup> was ascribed to the stretching vibration absorption peak of hydroxyl (—OH) in the sugar unit. The C—H stretching vibration absorption peak at 2925 cm<sup>-1</sup> was from methyl or methylene (Shao *et al.* 2011). The absorption peak at 1640 cm<sup>-1</sup> was due to water absorbed by hydroxyls on the molecule chains (Chen *et al.* 2013). The strong absorption peak at 1415 cm<sup>-1</sup> was produced by the symmetrical stretching vibration of the glycuronate —COO<sup>-</sup>, confirming the presence of the glucuronic acid (Peng 2010). The stretching vibration of the C—C bond accounted for the peak centered at 1262 cm<sup>-1</sup>. The peak at 1044 cm<sup>-1</sup> was the typical absorption peak of hemicellulose, which could be ascribed to the stretching vibration of C—O and C—C bonds or the bending vibration of C—OH (Chaikumpollert *et al.* 2004; Sun *et al.* 2004). The characteristic absorption peaks at 897 cm<sup>-1</sup> indicated that  $\beta$ -glycosidic bonds were the connections of xylose units within poplar hemicellulose (Sun and Tomkinson 2002). The characteristic peak of lignin 1506 cm<sup>-1</sup> did not appear, implying that the extracted hemicellulose contained little or no lignin (Sun *et al.* 1998).

The chemical structure of poplar hemicellulose was further characterized by <sup>1</sup>H-NMR (Fig. 6), and the results of the chemical shift assignment are listed in Table 6. The signal peak generated by D<sub>2</sub>O was at 4.75 ppm, and the peak at 4.4 ppm to 5.3 ppm represented the signal peak of the end proton ( $\alpha$  configuration 5.0 ppm to 5.3 ppm,  $\beta$  configuration 4.4 ppm to 4.6 ppm) (Kormelink *et al.* 1993; Kardošová *et al.* 1998; Teleman *et al.* 2000; Chiarini *et al.* 2004; Moine *et al.* 2007; Nabarlatz *et al.* 2007). The signal peaks at 4.44 ppm, 3.55 ppm, 3.72 ppm, and 4.08(3.35) ppm that represented C<sub>1</sub>—H, C<sub>3</sub>—H, C<sub>4</sub>—H, and C<sub>5</sub>—H keys of  $\beta$  (1→4) connected to xylose. The peak at 3.42 ppm belonged to the methyl protons signal peak of 4-*O*-methyl glucuronic acid, illustrating that 4-O-methyl glucuronic acid was substituted for the C<sub>2</sub> position of poplar hemicellulose. Meanwhile, its main C<sub>4</sub>—H and C<sub>1</sub>—H signal peaks were found at 3.17 ppm and 5.22 ppm. The <sup>1</sup>H-NMR results corresponded to the IC and FT-IR analysis.



Fig. 6. The <sup>1</sup>H-NMR spectrum of extracted poplar hemicellulose

Sugar Majaty	Chemical shift (ppm)							
Sugar Molety	C₁-H	C <sub>2</sub> -H	C₃-H	C <sub>4</sub> -H	C <sub>5</sub> -H <sup>ax</sup>	C <sub>5</sub> -H <sup>eq</sup>	OCH <sub>3</sub>	
$\beta$ (1 $\rightarrow$ 4) xylose 4.44 nf 3.55 3.72 3.34 4.08 -								
4-O-methyl glucuronic acid 5.22 nf nf 3.17 nf - 3.42								
C <sub>5</sub> —H <sup>ax</sup> : hydrogen on the axial bond; C <sub>5</sub> —H <sup>eq</sup> : hydrogen on the equatorial bond; nf: no obvious								
signal peaks								

	Table 6.	Chemical	Shift Assign	nment of E	Extracted F	Poplar	Hemicellulose
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#### Thermal properties of hemicellulose

The thermal stability of poplar hemicellulose was evaluated by thermogravimetric analysis (TGA), and the TGA/DTG curves of poplar hemicellulose are shown in Fig. 7. The thermal weight loss of hemicellulose mainly occurred in the stages of 40 °C to 200 °C, 200 °C to 350 °C, and 350 °C to 600 °C. The weight loss between 40 °C and 200 °C was caused by evaporation, which was also confirmed with the water absorption peak of 1640 cm<sup>-1</sup> in the FT-IR spectrum (Sun and Tomkinson 2002). The main weight loss stage of hemicellulose occurred in the range of 200 °C to 350 °C, with the corresponding initial decomposition temperature of 214 °C and the thermal decomposition rate peak temperature of 251 °C. At this stage, the C—O, C=O, and other bonds on the polymer side chains cracked, which produced large quantities of gas such as steam, carbon monoxide, carbon dioxide, methane, and acetic acid (Shukry *et al.* 2008). When the temperature exceeded 350 °C, the thermal decomposition rate of hemicellulose was prominently reduced and entered the carbonization process, namely the C—C main chains on the polymer skeleton cracked accompanied by the production of some flammable gases (Soliman *et al.* 1997; Yang *et al.* 2012).



Fig. 7. TGA/DTG curves of extracted poplar hemicellulose

#### CONCLUSIONS

- 1. The maximum extraction yield of hemicellulose obtained with Box-Behnken design was 52.8% for the optimum extraction parameters by RSM analysis (NaOH concentration of 9.5%, reaction time of 4 h 12 min, and temperature of 78 °C).
- 2. Under the optimal condition, alkaline extraction was less damaging to the structure of hemicellulose. Moreover, the obtained hemicellulose had a high degree of polymerization and a low degree of branching. The FT-IR and TGA analysis demonstrated excellent water absorption and heat resistance of hemicellulose, which were conducive to subsequent processing and utilization.
- 3. This efficient extraction of hemicellulose is an alternative and promising process for transforming poplar residue into chemicals of high utilization value in papermaking, food packaging, and biomedical fields. Development of technology to modify and exploit poplar hemicellulose, to make advantageous use of its good biodegradability, barrier performance, and heat resistance, should become the focus of future research.

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