

# Kinetic Model Analysis of Mild Autohydrolysis of Eucalyptus Woodchips for Xylose Production

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The autohydrolysis process of eucalyptus hemicelluloses was studied. The effect of hydrolysis temperature and soaking time on the dissolution of hemicellulose from eucalyptus chips was considered, and the process conditions were further optimized by *P* factor. The kinetic equations for the hemicellulose reaction were derived by studying the reaction process of hemicellulose during the pre-hydrolysis of eucalyptus chips, based on the reaction activation energy of xylose from eucalyptus chips and the investigated reaction mechanism of eucalyptus pre-hydrolysis process. The results demonstrated the feasibility of efficient extraction and utilization of hemicellulose in the process of dissolving pulp preparation from eucalyptus chips.

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## INTRODUCTION

In the traditional process of pulp and papermaking, a considerable part of hemicellulose is dissolved from the fibrous raw material into the black liquor and eventually burned together with lignin in the alkali recovery process. Hemicellulose may not be fully used during the burning process, because its calorific value is only about half of that to lignin (Duan *et al.* 2013). The utilization of hemicellulose has been mainly focused on isolation process, structural analysis, and transformation technology for conversion into bioenergy and value-added chemicals, expanding the utilization of hemicellulose for its industrial application (Mudoga *et al.* 2008; Yu *et al.* 2008; Bian *et al.* 2013; Azelee *et al.* 2016). Among them, the study of xylo-oligosaccharides (XOS) has attracted much interest. However, XOS currently is prepared solely based on corn cob, its production and costs are greatly affected by the source of the raw material. Furthermore, pollution from the massive solid waste produced during production is another problem (Yang *et al.* 2005). Therefore, to avoid that restriction of raw material and the consequential issues caused by XOS production, the integration of XOS production into the existing process for practical application has been suggested. Pre-hydrolysis during dissolving pulp production is a separation process of hemicellulose, and the main component of hardwood hemicellulose is xylo-oligosaccharides, which offers a favorable opportunity for the simultaneous production of XOS and dissolving pulp.

In the natural plant structure, hemicellulose covers the surface of cellulose microfibril connected by hydrogen bonds, and hemicellulose is connected with lignin by covalent bonds to form the water-insoluble structure (Yan *et al.* 2014). During the biorefining process, cellulose and hemicellulose need to be separated effectively by opening the molecular chains of plant fiber-dense structures and polymers. Among the numerous separation processes, the pre-hydrolysis kraft (PHK) process, also known for dissolving pulp production, has been used to selectively remove the hemicellulose. Pre-hydrolysis performed by adopting solely hot water or steam, also known as autohydrolysis, is mainly affected by the acetic acid released by the unstable acetyl groups present in the hemicellulose. The extent of xylan hydrolysis depends on the amount of hardwood species, maximum temperature of pre-hydrolysis, soaking time, pH, and liquid-to-solid ratio (Fang *et al.* 2014). The dynamics of the wood components are essential to understand to control the performance of the pre-hydrolysis during the PHK process, by establishing a balanced relationship between the pre-hydrolysis severity and delignification efficiency under given subsequent cooking conditions (Zhan *et al.* 2011). Moreover, it is complex for polysaccharide hydrolysis in plant fiber raw materials, during which process so many physicochemical reactions occur. Studying the hydrolysis properties of polysaccharides is helpful in understanding the whole process of hydrolysis.

In this study, eucalyptus hemicellulose was hydrolyzed at different temperatures and periods of time. The reaction process of hemicellulose during the pretreatment of eucalyptus in hot water was studied. The reaction kinetic equation of hemicellulose was established to get the reaction activation energy of xylose. The pre-hydrolysis reaction law of eucalyptus hemicellulose was explored to provide support for the efficient extraction and utilization of hemicellulose in the process of dissolving pulp preparation from eucalyptus chips.

## EXPERIMENTAL

### Materials

Eucalyptus wood chips sampled from a pulp mill in Jining City (Shandong Province, China) were selected and stored in a sealed plastic bag. Chemical components of eucalyptus are listed in Table 1.

**Table 1.** Main Chemical Components of Eucalyptus

Chemical Components	Content (%)
Ash	0.50
Benzene-alcohol extractive	1.26
Holocellulose	80.23
Pentosan	23.85
Acid insoluble lignin	21.08
Acid-soluble lignin	2.40
Total lignin	23.48
Glucose	48.72
Xylose	18.07
Arabinose	0.31
Galactose	1.79
Mannose	2.13

## Instruments and Equipment

The main instruments and equipment used in the experiment were as follows: a Model I wood chip grinder (CNPPRI, Beijing, China); an HH. S11-Ni6 electric-heated water-bath (328 Scientific Instruments Co., Ltd., Beijing, China); a MS204S/01 analytical balance (Mettler Toledo, Columbus, OH, USA); a AU/E-20 digester (RegMed Corporation, Osasco, Brazil); a UV-1800 UV-visible spectrophotometer (Shimadzu Corporation, Kyoto, Japan); a FD-1D-50 freeze drier (Beijing Bokang Experimental Instrument Co., Ltd., Beijing, China); a DIONEX ICS-3000 high performance liquid chromatograph (Agilent Technologies Co., Ltd., Santa Clara, CA, USA); a Hitachi 7000 chromatograph of gel permeation (Hitachi Co., Ltd., Tokyo, Japan); and a 769YP-15A powder compressing machine (Tianjin Tech High-tech Company, Tianjin, China).

## Analysis of the Chemical Components of Eucalyptus Raw Materials

Eucalyptus woodmeal of size 40- to 60-mesh was collected in a sealed plastic bag after the eucalyptus chips were ground and screened. The content of ash, benzene-alcohol extractive, holocellulose, pentosan, acid insoluble lignin, and acid-soluble lignin were tested according to the following standards: TAPPI T211 om-07 (2007), GB/T 2677.6 (1994), GB/T 2677.10 (1995), GB/T 2677.9 (1994), GB/T 2677.8 (1994), GB/T 10337 (1989), respectively. The biomass components of raw material were determined according to the standard methods developed by the US National Laboratory for Renewable Energy (Sluiter *et al.* 2008).

## Pre-hydrolysis Process of Eucalyptus

Pre-extraction of hemicellulose (sugar) from eucalyptus chips was conducted in a laboratory- scale digester according to the following process: samples of 100 g (oven-dried weight) eucalyptus chips with deionized water (liquor to wood ratio, L/W 1:6) were treated at the same heating-up time of 60 min from 35 °C to reach a different final temperature of 130, 150, 160, and 170 °C for various soaking times. The initial pH was 7.8. Specific conditions are shown in Table 2.

**Table 2.** Pre-hydrolysis Conditions of Eucalyptus Chips

Maximum Temperature (°C)	Process Conditions	Soaking Time (min)
130	L/W 1:6 heating-up time 60 min	0, 30, 45, 60, 75, 90, 105, 120, 150
150		
160		
170		

After pre-hydrolysis, the small reaction tank was removed and cooled in the cold-water bath for 10 min, then the hydrolyzed woodchips were filtered with a 300-mesh cloth bag to collect the pre-hydrolysis liquor for further analysis. The woodchips were washed, air dried, and stored into a self-sealed plastic bag for further analysis.

## P Factor

The hydrolysis temperature and time have remarkable effect on the extraction of hemicellulose. The pre-hydrolysis factor (or *P* factor) that can combine the two factors has been introduced to control and evaluate the hemicellulose removal reaction. Sixta (2006) provided a formula for the *P* factor calculation (Eq. 1) for blue eucalyptus pre-hydrolysis

based on the activation energy of carbohydrate glycoside bond breakage, assuming that reaction rate was 1 at 100 °C. The  $P$  factor calculation is as follows,

$$P = \int_0^t \frac{V(T)}{V_{373K}} = \int_0^t \exp\left(40.48 - \frac{15106}{T}\right) dt \approx \frac{t_1}{360} \left[ \exp\left(40.48 - \frac{15106}{T_0}\right) + 4 \exp\left(40.48 - 2 \times \frac{15106}{T_0 + T}\right) + \exp\left(40.48 - \frac{15106}{T}\right) \right] + \frac{t_2}{60} \left[ \exp\left(40.48 - \frac{15106}{T}\right) \right] \quad (1)$$

where  $V(T)$  is the reaction rate at  $T$  (K),  $V_{373K}$  is the reaction rate at 373 K,  $T_0$  is the initial temperature (K),  $T$  is the temperature at any moment (K),  $t_1$  is the time required from  $T_0$  to  $T$  (min), and  $t_2$  is the retention time (min).

The total  $P$  factor, including the sum of the  $P$  factor of the heating phase and the isothermal phase, is suitable to control the degree of purification during the preparation of dissolving pulp by the pre-hydrolytic kraft process (Feng and Huang 2011; Shan 2012). Therefore, the total  $P$  factor was applied in this study.

### Kinetic Analysis of Xylose Preparation from the Hemicellulose

In the autohydrolysis process, hydrolysis kinetics are characterized by the rate at which hemicellulose hydrolysis produces monosaccharides (Lu *et al.* 2008). The analysis of oligosaccharides and their introduction into the hydrolysis dynamics of hemicellulose remains the focus of hydrolysis dynamics studies. Assuming that the dynamics of xylan is fully in contact with water, no restriction affects its diffusion rate, and the xylan dissolution rate is proportional to the xylan concentration, the dynamics of xylan should follow the first-order kinetic reaction equation. Meanwhile, xylans may contain parts of different reactivity that could affect the reaction rate. It was found that the reaction rate was noticeably slowed down at approximately 70% conversion rate. As proposed by Veeraraghavan in 1982 and Conner in 1984, xylan is composed of two-parts: fast-xylan and slow-xylan, each part of which followed the first-order kinetic reaction equation (Belkacemi *et al.* 1991; Carrasco and Roy 1992), and the rate of xylan hydrolysis was expressed as Eq. 2. The model was based on the presence of two types of xylans that followed a parallel first-order reaction hydrolysis,

$$X = Z_X \times \text{Exp}(-k_{f,X} \times t) + (1 - Z_X) \times \text{Exp}(-k_{s,X} \times t) \quad (2)$$

where  $X$  is the fractions of original xylans remaining in the solid residue,  $Z_X$  is the fractions of the readily reactive original xylan remaining in the solid residue,  $k_{f,X}$  is the reaction constant of the fast-xylan hydrolysis,  $k_{s,X}$  is the reaction constant of slow-xylan hydrolysis, and  $t$  is the soaking time.

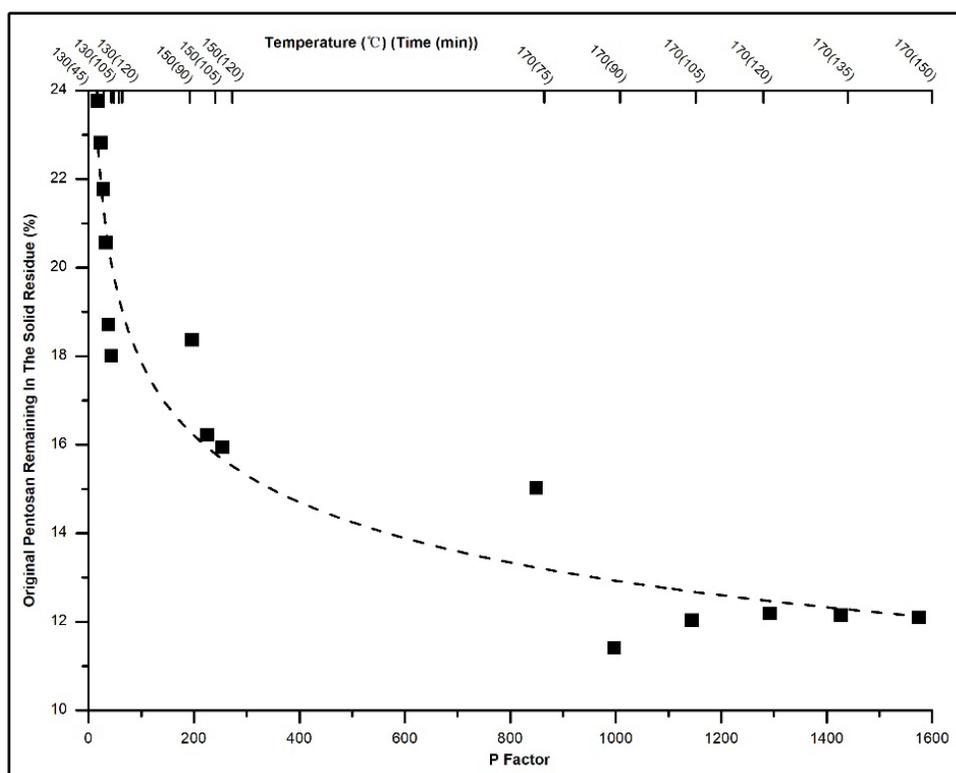
The acid concentration was unchanged in the higher liquid ratio during the hydrolysis process. The pre-hydrolysis reaction kinetics of eucalyptus chips at a liquid to wood ratio of 1:6 was the focus of this study.

## RESULTS AND DISCUSSION

### Residual Pentosan Content in Eucalyptus Chips After Pre-hydrolysis

The purpose of the hydrolysis is to maximize hemicellulose hydrolysis, while avoiding further hydrolysis into single sugars. The study of the residual pentosan content in eucalyptus chips after water pre-hydrolysis was to indicate the degree of hemicellulose

hydrolysis. As shown in Fig. 1, with the  $P$  factor ranging from 18 to 1575, the dissolution of pentosans was divided into two stages: a large dissolution stage and a residual dissolution stage. Pentosans dissolved rapidly from eucalyptus chips when the  $P$  factor was less than 1000, with the dissolution rate over 52.00% at the pre-hydrolysis temperature of 170 °C and a soaking time of 90 min. Thereafter, the dissolution rate was noticeably slower. When the soaking time was extended to 150 min, only 0.69% of the pentosan was additionally dissolved. Unlike in the previous stage, the structure and performance of the pentosan dissolved at this stage were mainly a class of pentosan macromolecules with high molecular weight and low reactive activity, which were much harder to be hydrolyzed for its complex internal structure, and closely connected with lignin and cellulose.



**Fig. 1.** The residual pentosan content in eucalyptus chips after water pre-hydrolysis under different conditions

### Branching Degree

Most linear hemicellulose molecular structures contain some short-branched chain structures. For hardwood, a higher ratio of glucuronate to xylose in the hemicellulose molecular structure resulted in a higher branching degree (Bian 2013). As displayed in Fig. 2, the branching degree of the hemicellulose in the pre-hydrolysis liquor was overall low. The branching degree of hemicellulose decreased with the increase of  $P$  factor, indicating that the side chains were easier to break at a higher  $P$  factor. When the  $P$  factor was increased to over 600, the final branching degree stayed at around 0.1, meaning that there was 1 glyoxylate branch chain on the backbone of every 10 xylose units. Furthermore, the disappearance of the hemicellulose glucuronate group in the pre-hydrolysis liquor illustrated the continuous degradation of the hemicellulose.

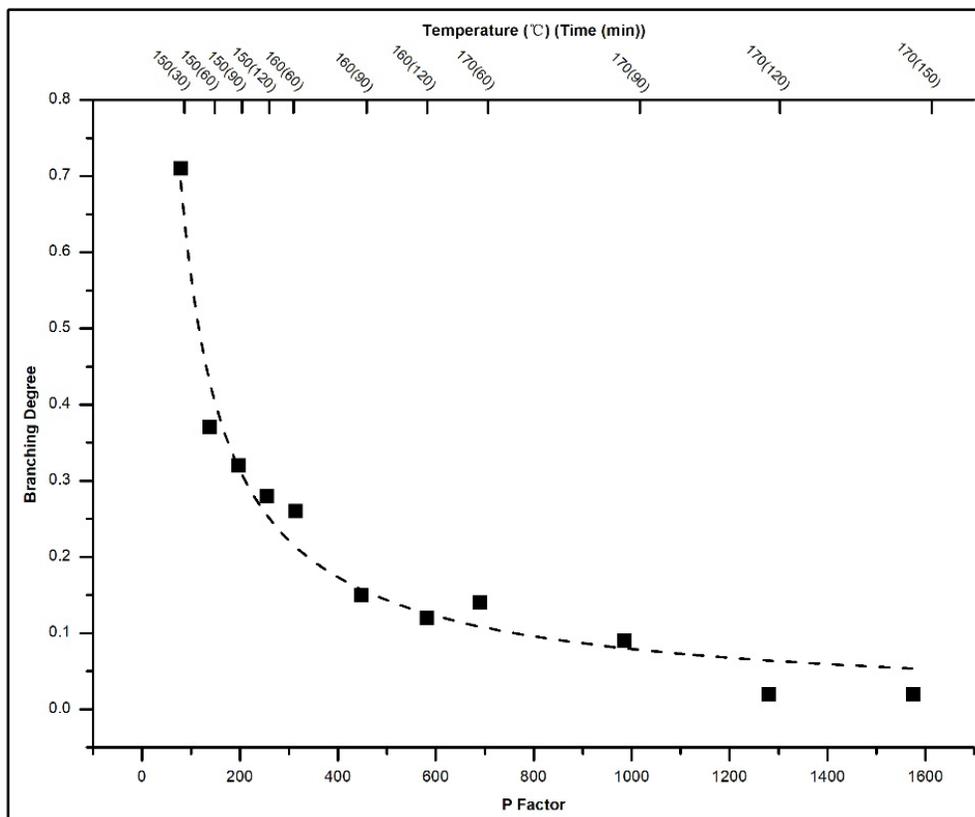


Fig. 2. The branch degree of the hemicellulose at different P factor

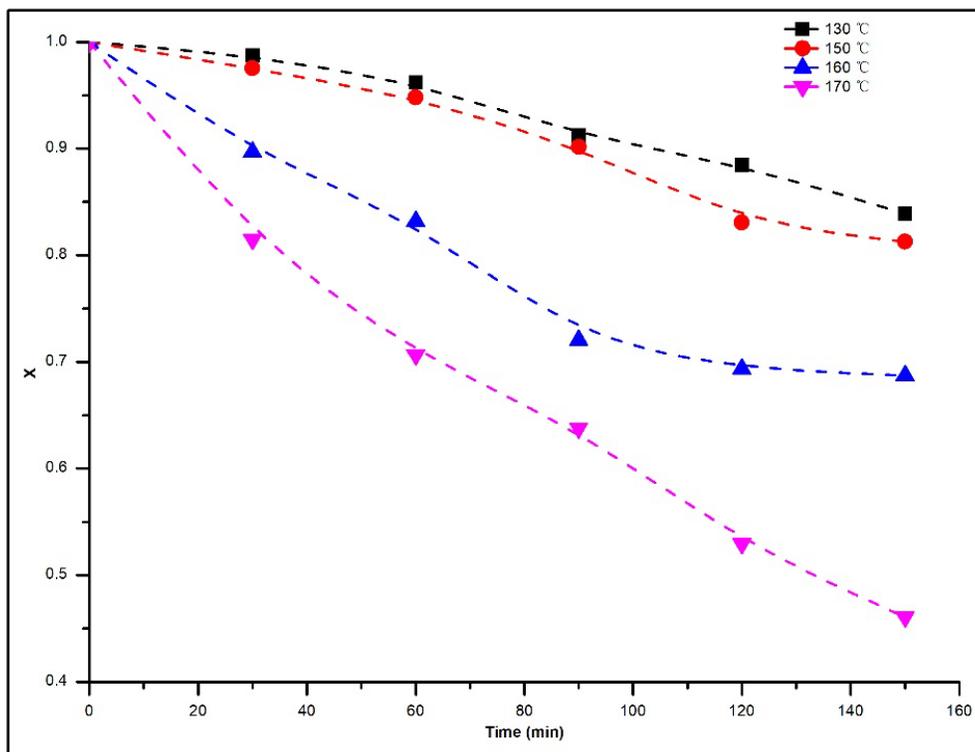


Fig. 3. The relation between dissolved hemicellulose and the temperature and time

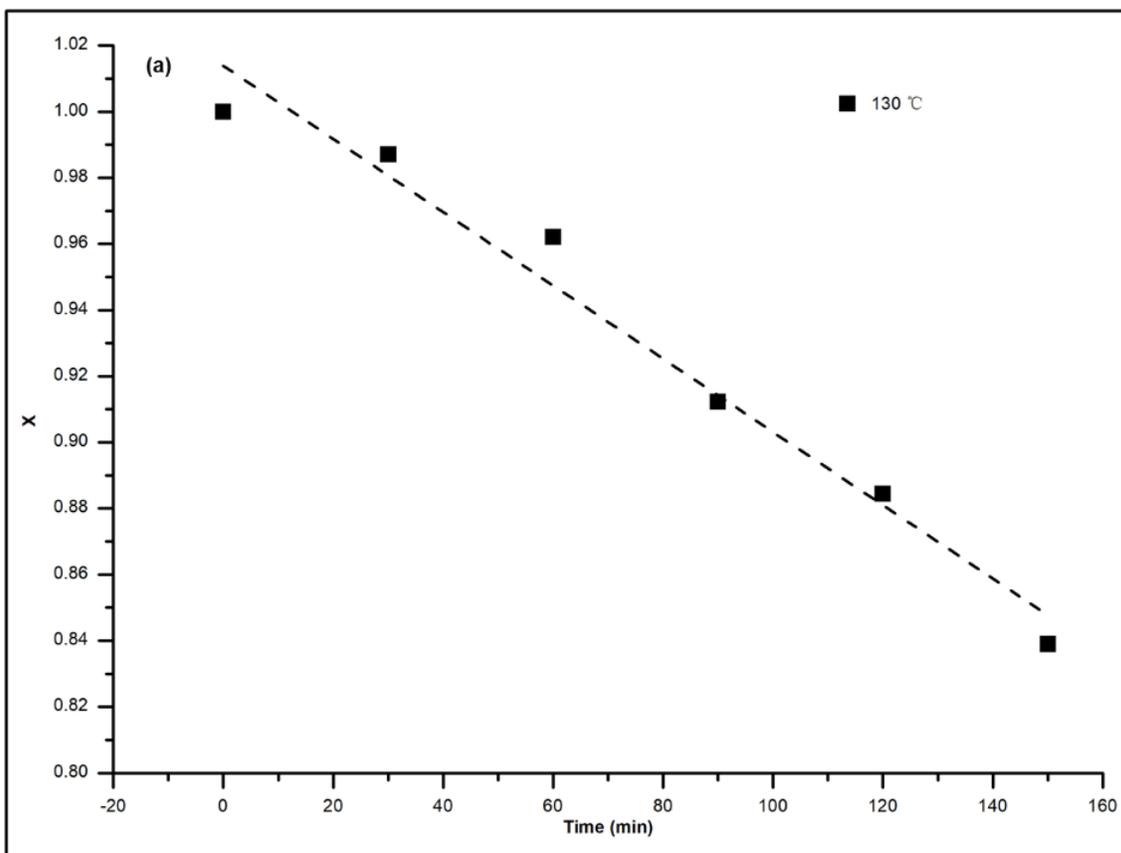
## Kinetics Analysis of the Hydrolysis from Hemicellulose to Xylose

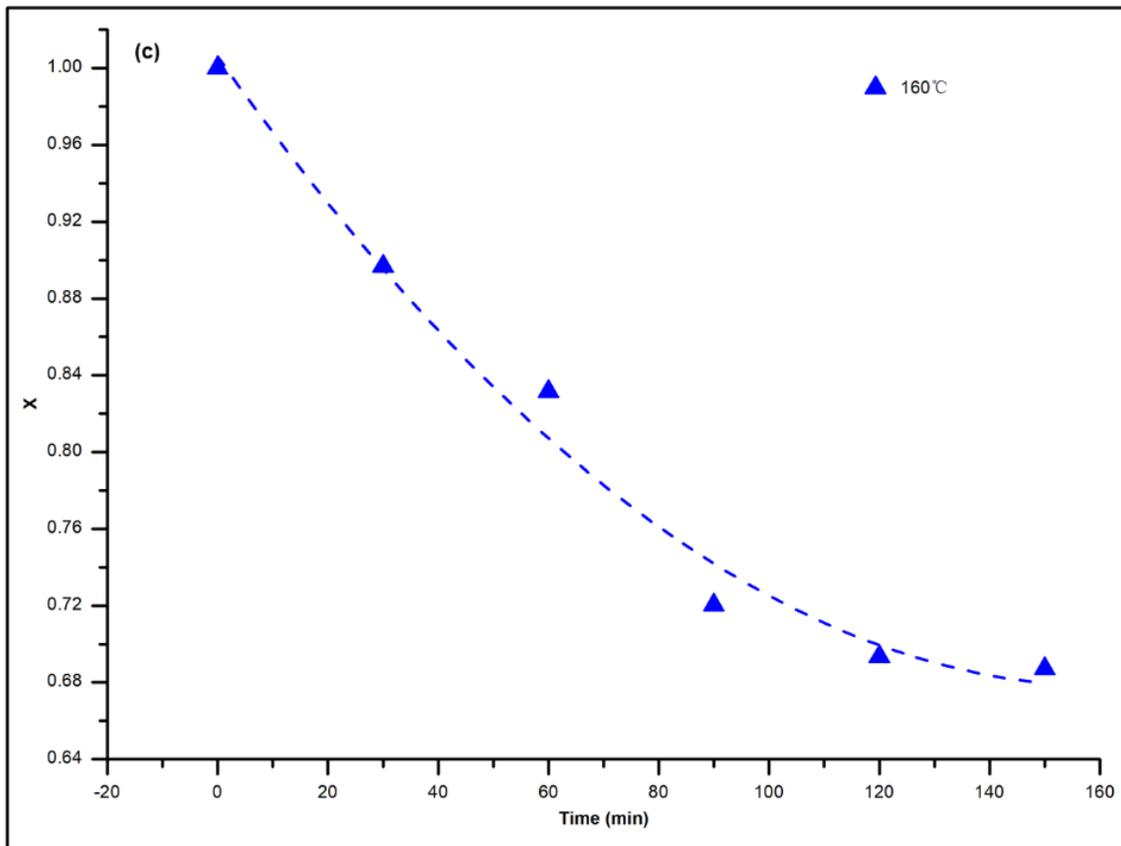
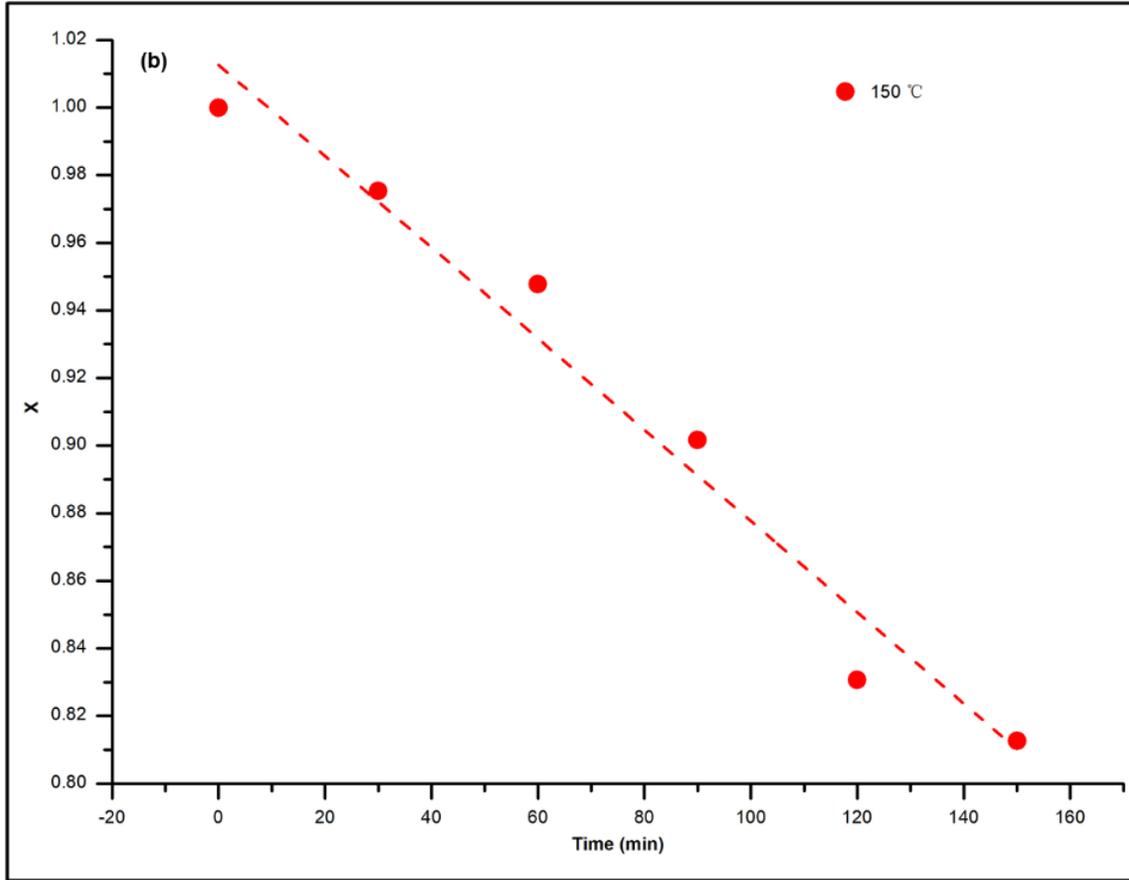
### *Experimental results of water pre-hydrolysis dynamics*

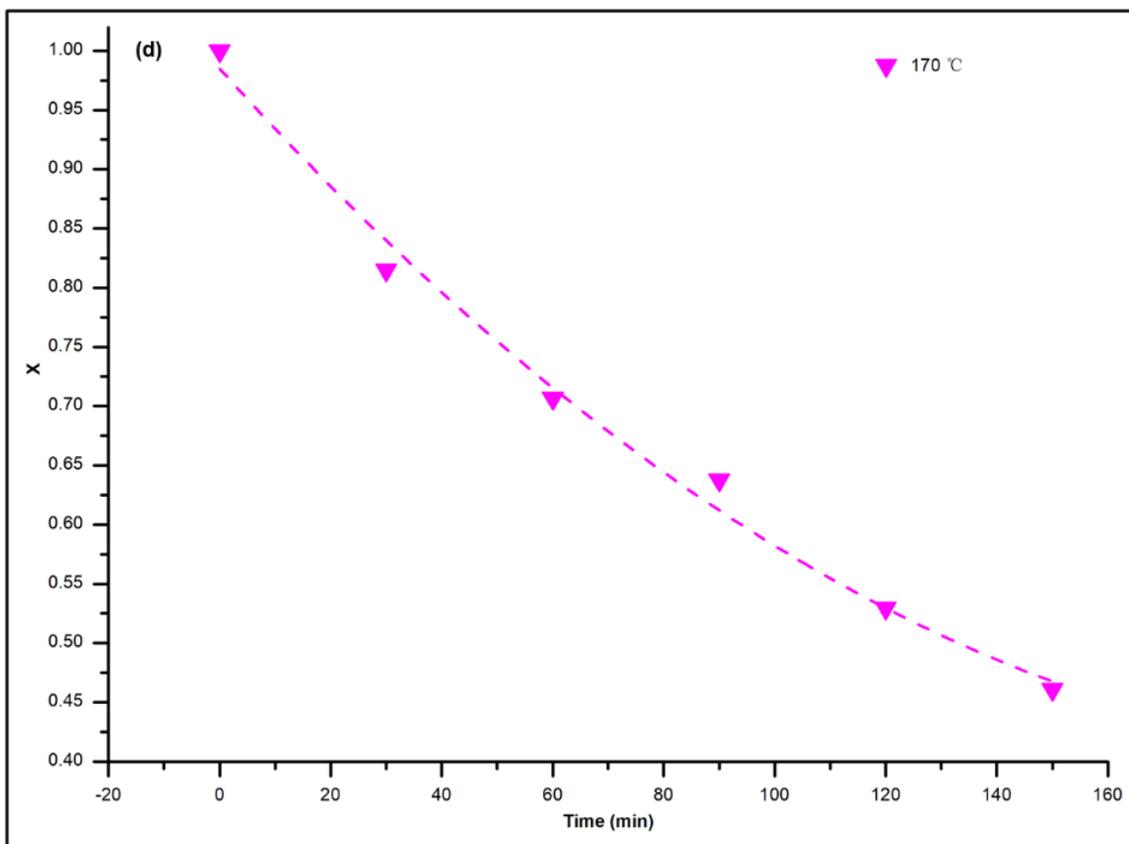
The extent of hemicellulose dissolution during water pre-hydrolysis was characterized by the ratio of residue xylose content in eucalyptus chips after pre-hydrolysis to original xylose content in the raw material, expressed as  $X$  (Fig. 3). As featured in Fig. 3, the hydrolysis temperature had a great effect on the hydrolysis of xylose. There was still 83.9% of xylose residue in the wood chips after being treated for 150 min at 130 °C. In contrast, when the hydrolysis temperature reached 170 °C, numerous hemicelluloses were hydrolyzed in a short time, showing a sharp reduction in the proportion of residual xylose in the wood chips, over 50% of the xylose were already dissolved within 150 min. Therefore, it was more suitable to adopt the high-temperature and short-time thermal insulation pre-hydrolysis process.

### Determination of the Reaction Rate Constant

The reaction rate constants  $k_{fX}$ ,  $k_{sX}$ , and  $Z_X$  were obtained by substituting the  $X$  value from Fig. 3 into Eq. 2 and using its nonlinear fit by Matlab software (The MathWorks Inc., Matlab 7.0, Natick, MA, USA). The results are shown in Fig. 4 and Table 4, where  $R^2$  was the correlation coefficient indicating the degree of fit.







**Fig. 4.** Nonlinear fit diagram of X and soaking time at different hydrolysis temperatures (a) 130 °C, (b) 150 °C, (c) 160 °C, and (d) 170 °C

**Table 4.** Kinetics Parameters of the Hydrolysis from Hemicellulose to Xylose and Its Fitting Results

Maximum Temperature (°C)	$k_{f,x}$ (min <sup>-1</sup> )	$k_{s,x}$ (min <sup>-1</sup> )	Zx	R <sup>2</sup>
130	0.0011	0.0009	0.6344	0.9441
150	0.0014	0.0013	0.5456	0.9481
160	0.0141	0.0010	0.2583	0.9762
170	0.0677	0.0039	0.0988	0.9993

Table 4 shows that the relationship was well fitted. The Zx decreased with increasing hydrolysis temperature, while  $k_{f,x}$  and  $k_{s,x}$  increased with increasing hydrolysis temperature, of which  $k_{f,x}$  increased more remarkably. It was shown that Zx mainly depended on the diffusion rate of the hydrolysate and hydrolysis products when the liquid-to-solid ratio was small, and Zx was mainly affected by the raw material structure and hydrolysis temperature when the liquid-solid ratio was relatively large (Tan *et al.* 2012). Increasing the liquid-to-solid ratio can promote the reaction rate.

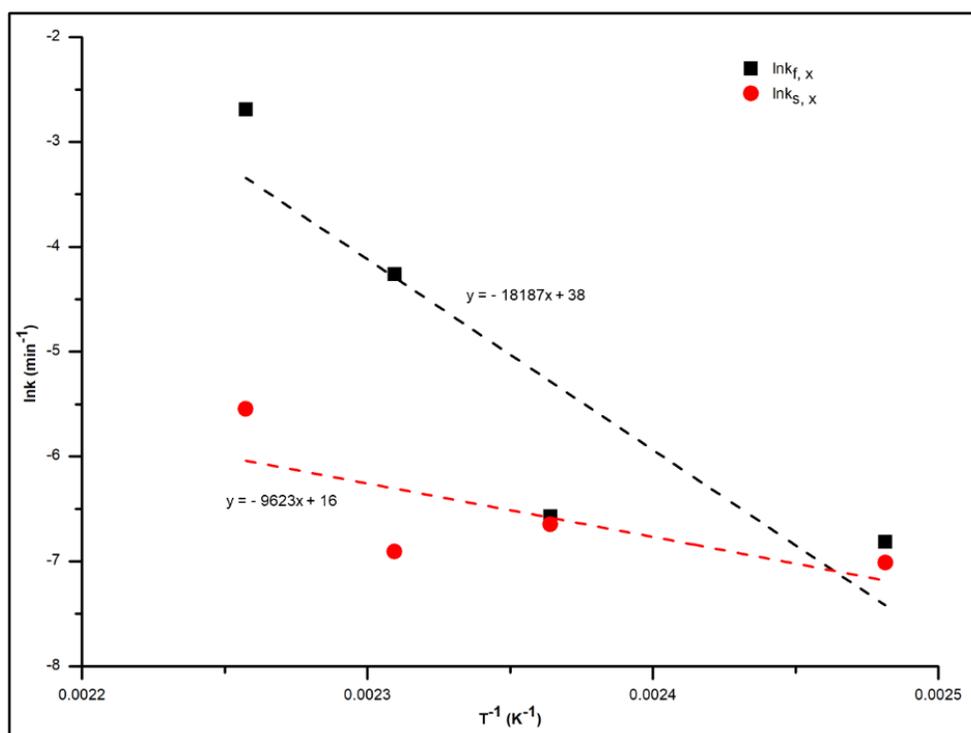
### Identification of the Activation Energy

According to the Arrhenius Equation (Eqs. 3 and 4),

$$k = A \times e^{-\frac{E_a}{kT}} \quad (3)$$

$$\ln k = -\frac{E_a}{R} \times \frac{1}{T} + \ln A \quad (4)$$

where  $A$  is the anterior factor,  $E_a$  is the reaction activation energy (kJ/mol),  $R$  is the gas constant (J/(mol K)); and  $T$  is the absolute temperature (K,  $T = T_1 + 273$ ) ( $T$  is the absolute temperature (K), and  $T_1$  is the Celsius temperature ( $^{\circ}\text{C}$ )).



**Fig. 5.** The fitted curve of the  $\ln k$  and  $1/T$

Activation energy can be derived by linear fitting of  $k_{fX}$  and  $k_{sX}$  to  $1/T$ , as shown in Fig. 5. The reaction activation energy was the slope of the line, so that the fast reaction activation energy  $E_{fX}$  and the slow reaction activation energy  $E_{sX}$  were 151 kJ/mol and 80 kJ/mol, respectively.

For eucalyptus water pre-hydrolysis, the fitting line slope varied remarkably with the temperature increasing from 130 to 170  $^{\circ}\text{C}$ , namely,  $E_{fX}$ , and  $E_{sX}$ , were quite different;  $E_{fX}$  was relatively high, while  $E_{sX}$  was low. The reaction speed was related to the temperature and the pressure, and the activation energy was a marker of temperature sensitivity. The reaction with large activation energy acted much faster when the temperature changed, even though its reaction rate was slower at normal conditions. Because the slow reaction determines the entire process of pre-hydrolysis, increasing temperature is the most efficient way to accelerate the degree of hydrolysis while ensuring no degradation of cellulose and fewer lignin condensation reactions.

Therefore, the reaction rate equation of eucalyptus water pre-hydrolysis from 130 to 170 °C can be derived by substituting the fitted kinetic parameters into the Arrhenius equation, as depicted in Eqs. 5 and 6:

$$k_{f,x} = 3.18 \times 10^{16} \times e^{-\frac{151}{RT}} \quad (5)$$

$$k_{s,x} = 8.89 \times 10^6 \times e^{-\frac{80}{RT}} \quad (6)$$

## CONCLUSIONS

1. Pentosans dissolved rapidly from eucalyptus chips when the  $P$  factor was less than 1000, with the dissolution rate over 52.0% at the pre-hydrolysis temperature of 170 °C and a soaking time of 90 min. Thereafter, the dissolution rate was remarkably slower. When the soaking time was extended to 150 min, only 0.69% of the pentosan was further dissolved.
2. The branching degree of hemicellulose decreased with the increase of the  $P$  factor, indicating the side chain was easier to break at a higher  $P$  factor. When the  $P$  factor was increased to over 600, the final branching degree was around 0.1, meaning that there was 1 glyoxylate branch chain on the backbone of every 10 xylose units.
3. The dissolution dynamics of xylan for eucalyptus water pre-hydrolysis from 130 to 170 °C followed the first-order kinetic reaction equation, and the hydrolysis temperature was the main factor affecting the degree of hydrolysis.
4. According to the Arrhenius equation, the fast reaction activation energy  $E_{f,x}$  and the slow reaction activation energy  $E_{s,x}$  of eucalyptus water pre-hydrolysis in the range of 130 to 170 °C were 151 and 80 kJ/mol, respectively.

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