REMOVAL OF HEMICELLULOSES BY NaOH PRE-EXTRACTION FROM ASPEN CHIPS PRIOR TO MECHANICAL PULPING

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Hemicelluloses can be removed from wood chips prior to mechanical pulping, which would offer new feedstocks for the production of chemicals and fuels. The aim of this study was to evaluate pre-extraction to maximize pre-extraction yield, while minimizing negative impacts on wood chips. The effects of three independent process variables (NaOH charge, pre-extraction temperature, and time) on three dependent variables (pre-extraction yield, xylan extraction yield, and cellulose content based on original wood) were studied using a Box-Behnken experimental design. The mathematical models were obtained and validated well. It was found that NaOH charge, time, interaction between NaOH charge and time, and interaction between temperature and time have significant effects on xylan extraction yield. The xylan extraction yield was 22.55%; i.e., about 37.3 kg of xylan could be extracted from one ton of oven-dried aspen chips under the conditions of 5.68% NaOH charge, 100 °C, and 35 min.

Keywords: Hemicelluloses; Removal; Alkali pre-extraction; Xylan extraction yield;

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INTRODUCTION

The concept of value prior to pulping (VPP) (van Heiningen 2006; Thorp and Raymond 2004) has been proposed, where the hemicelluloses are either partially or completely extracted for biofuel production. The remaining solids (mainly cellulose and lignin) can be further delignified for wood pulp or fiber production (Zhu and Pan 2010). Such pre-extraction of hemicelluloses would potentially mesh will with efforts to convert current pulp mills into future integrated biomass refineries (Gullón et al. 2010). The extracted hemicelluloses could be transformed into biofuels, biochemicals, and biomaterials, such as bioethanol, biohydrogen, furfural, xylitol (Parajo et al. 1998), bifunctional organic molecules, barrier films (Grondahl et al. 2004) and hydrogels (Lindblad et al. 2001). The hemicelluloses released during the pulping process would accumulate and increase the effluent load and then contribute to the post-treatment cost in a papermaking process (Johnsen and Stenius 2007; Svedman et al. 1995). Therefore, pre-extraction of hemicelluloses from wood chips prior to pulping not only can offer new feedstocks for the production of chemicals and fuels but also would remedy some of the operational problems in pulp and paper mills (Boluk et al. 2008). In addition, extracting hemicelluloses prior to pulping can also benefit the energy recovery from chemical pulping liquors, because the heating value of hemicelluloses is about one half of that of lignin (van Heiningen 2006).

So far, all the research related to the VPP concept have been focusing on chemical pulping processes (Al-Dajani and Tschirner 2008; Helmerius et al. 2010; Yoon et al. 2008) and few attempts have been applied to mechanical pulping processes. The chemical pretreatment stage in chemi-mechanical pulping process could be easily retro-fitted to serve as the pre-extraction stage without major capital investment. However, due to the significant advantages of mechanical or chemi-mechanical pulping, (e.g., high pulping yield), the severe conditions used in chemical pulping process, such as high chemical charge, high liquid/wood ratio, long duration time, and high temperature, are not suitable any more. So, it is very necessary to do some studies on pre-extraction to balance the potential extraction yield and degradation of wood chips.

In this study the hemicelluloses were pre-extracted with alkaline peroxide in the vapor phase of an M/K digester. Alkaline pretreatment has been widely used in the pretreatment of biomass (Sun et al. 2000; Bjerre et al. 1996; Gabrielii et al. 2000). It can be carried out at lower temperatures and lower pressures than other pretreatment methods (Mosier et al. 2005; Carvalheiro et al. 2008). It has been proven that the alkaline pretreatment would be more suitable for hemicelluloses extraction from hardwoods than softwoods (Simonson 1965). For the alkaline peroxide extraction, it was found that the addition of hydrogen peroxide can promote the extraction yield of hemicelluloses to some extent (Fang et al. 2000). Therefore, a small amount of hydrogen peroxide has been used in this study. The aim of this study was to investigate the effects of process conditions (NaOH charge, pre-extraction temperature, and time) on hemicelluloses pre-extraction and wood chips properties to maximize hemicelluloses extraction efficiency, while minimizing degradation of wood chips. The hemicelluloses content was determined as xylan content in this study.

EXPERIMENTAL

Materials

Aspen chips from an eastern Canadian mill were screened before the pre-extraction. The main chemical components of the aspen chips were as follows: glucan 47.07%, xylan 16.50%, acid soluble lignin 3.54%, Klason lignin 17.93%, and acetone extractives 1.07%. The average errors for glucan, xylan, lignin, and extractives were 1.92%, 0.91%, 0.41% and 0.01%, respectively. NaOH, H_2O_2, and diethylene triamine penlaacetic acid (DTPA) used in the experiment were all of industrial grade. Acetone and H_2SO_4 used for acetone extractives and chemical components analysis were of analytical grade.

Methods

Chemicals Impregnation

The screened aspen chips were presteamed at 100 °C for 15 minutes in a chip bin. The impregnation of pre-extraction chemicals into the wood chips was carried out with an Andritz-Bauer 6’’ MSD press impregnator (Andritz Technology Ltd., Canada) (Yuan et al. 2006; Chagaev et al. 2005). The press impregnator is a typical process unit in chemimechanical pulping to ensure uniform and effective penetration of chemicals into wood chips. The concentrations of NaOH used in the impregnation were 1%, 3%, and 5% (w/v), which resulted in an NaOH charge on wood of 1.45%, 3.85%, and 5.68% (w/w), respectively. The ratio of $NaOH/H_2O_2$ and the DTPA charge were fixed at 2 and 0.2%, respectively.

Pre-extraction in the M/K digester

In this study, the pre-extraction was conducted in vapor phase of an M/K digester. The Liquid/Wood ratio (L/W) in this study was about 2:1. The heating procedure of the M/K digester (model 409, M/K system Inc., USA) was automatically controlled with a PLC controller. When the pre-extraction was finished, the pressure was released from the top valve, and cold deionized water was directly and immediately injected into the reaction vessel to terminate the reaction at high temperature. The extracted chips were taken out from the vessel. The liquor was collected from the bottom value. The extracted chips were broken down with a Waring blender to simulate a refining process (Shaw 1984; French and Maddern 1994; Chang et al. 2010). Then the extracts were obtained by filtering the extracted and blended chips slurry. The blended chips were washed thoroughly with deionized water and collected for further analysis. The cellulose content was determined as the glucan content based on original wood chips. The equation for the pre-extraction yield was as follows,

$$\text{Pre-extraction yield}=\left(1-\frac{m_1}{m_0}\right)\times 100\% \qquad (1)$$

where m_0 is the weight of oven-dried chips before pre-extraction and m_1 is the weight of oven-dried extracted chips.

The xylan extraction yield can be calculated with the following equation,

$$\text{Xylan extraction yield}=\frac{X_m - X_r}{X_m}\times 100\% \qquad (2)$$

where X_m is the xylan content of the aspen chips without pre-extraction and X_r is the xylan content of the extracted chips.

Chemical components analysis of chips and extract

Both the aspen chips and the extracted chips were air dried, milled, passed through a 20 mesh screen, and then were kept in a sealed plastic bag for further analysis. The content of extractives was determined according to the Pulp and Paper Technical Association of Canada (PAPTAC) standard procedures G.13 and G.20. The lignin content (acid soluble lignin and Klason lignin) was measured following PAPTAC standard procedures G.8 and G.9. The sugar analysis of aspen chips was performed with a Dionex DX-600 Ion Chromatograph system (Dionex, Sunnyvale, CA) according to the method described by Zhang (2009). These samples used for sugar analysis were filtrated through a 0.45 μm Millipore filter before analysis.

Experimental design

A Box-Behnken design (BBD) with three independent variables and three dependent variables was used to investigate the effects of NaOH charge, pre-extraction temperature, and time on pre-extraction yield, xylan extraction yield, and cellulose content based on original wood. Table 1 lists the conditions used in the study. Relatively mild conditions were used to minimize the degradation of wood chips.

RESULTS AND DISCUSSIONS

Mathematical Models

Table 2 lists the operational conditions corresponding to the experimental design, comprising of 15 sets of conditions, including 3 center points with random placement, as well as the results. The standard errors of the estimate show the standard deviation of the residuals for pre-extraction yield, xylan extraction yield, and cellulose content based on original wood are 1.1%, 1.5% and 0.4%, respectively. To investigate the effects of these three independent variables, including NaOH charge (A), pre-extraction temperature (B) and time (C), two main mathematic models were obtained and expressed in Equations (3) and (4):

$$\text{Pre-extraction yield} = -12.2966 + 1.0156A + 0.4929B + 0.1074C - 0.1172A^2 + 0.0073AB + 0.0343AC - 0.0036B^2 + 0.0002BC - 0.0030C^2 \quad (3)$$

$$\text{Xylan extraction yield} = -42.3590 + 7.2753A + 0.5413B + 0.9691C - 0.0528A^2 - 0.0231AB - 0.0714AC - 0.0017B^2 - 0.0046BC - 0.0030C^2 \quad (4)$$

Table 1. Box-Behnken Experimental Design

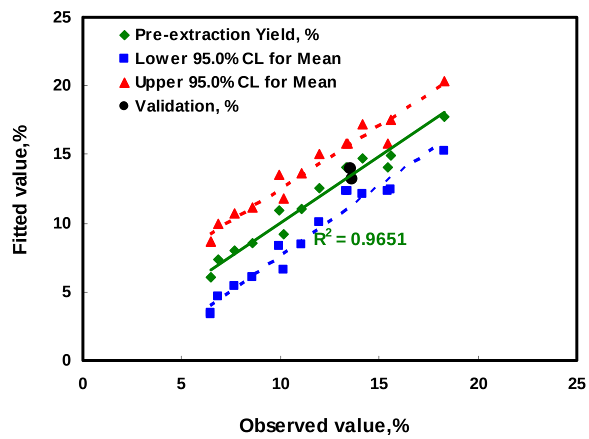
Independent variables		Units	Value or range
Independent variables	NaOH charge (A)	%	1.45, 3.85, 5.68
	Temperature (B)	°C	50-100
	Duration time (C)	min	10-60
Fixed conditions	NaOH /H_2O_2		2
	DTPA	%	0.2
Dependent variables	Pre-extraction yield	%	
	Xylan extraction yield	%	
	Cellulose content based on original wood	%	

Validation of the Models

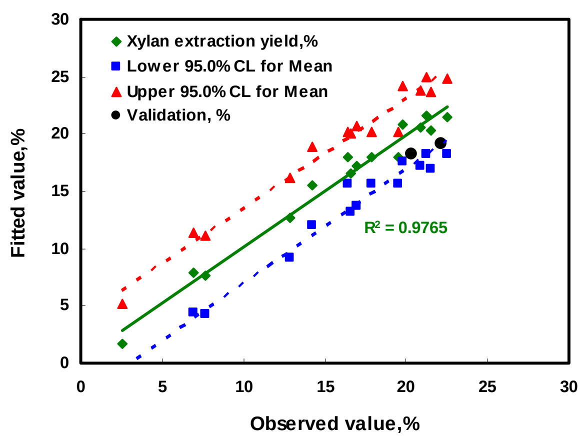
To validate the models, another two assays were performed under the following conditions: (1) 3.98% NaOH, 80 ºC, 60 min; (2) 3.98% NaOH, 75 ºC, 35 min. The results showed that all these calculated numbers were in good agreement with the experimental results, as shown in Fig. 1.

Table 2. The Conditions and Results Obtained from Experimental Design

NaOH, %	Temperature °C	Time, min	Pre-extraction yield, %	Xylan extraction yield, %	Cellulose content, %
1.45	75	60	6.45	16.95	46.90
1.45	100	35	6.50	12.83	46.86
5.68	50	35	14.12	21.24	43.74
3.85	75	35	13.40	19.47	44.37
3.85	100	60	9.94	16.62	45.80
3.85	100	10	8.55	14.22	46.45
3.85	50	10	10.17	7.63	45.91
5.68	75	10	12.01	20.87	45.98
5.68	100	35	15.57	22.55	44.00
3.85	50	60	11.10	21.54	45.99
3.85	75	35	13.34	16.44	44.53
3.85	75	35	15.42	17.88	44.43
1.45	50	35	6.86	6.96	46.50
1.45	75	10	7.65	2.58	46.99
5.68	75	60	18.31	19.81	43.51



(a)



(b)

Fig. 1. Relationships between observed and predicted values for pre-extraction yield (a) and xylan extraction yield (b)

Effects of Process Conditions on Pre-extraction Yield

The pre-extraction yield was regarded as one of the parameters to reflect the pre-extraction potential of hemicelluloses from aspen chips. The pre-extraction yield ranged from 5% to 19% in this study. The results showed that NaOH charge had the most significant effect on the pre-extraction yield, as shown in Fig. 2. The effects of two variables (NaOH charge and temperature) and their interactions on pre-extraction yield were depicted in the 3D response surface while the time was fixed at 35 minutes, as shown in Fig. 5(a). The results showed that with increase of NaOH charge, the pre-extraction yield increased significantly. With the increase of temperature, pre-extraction yield increased slightly.

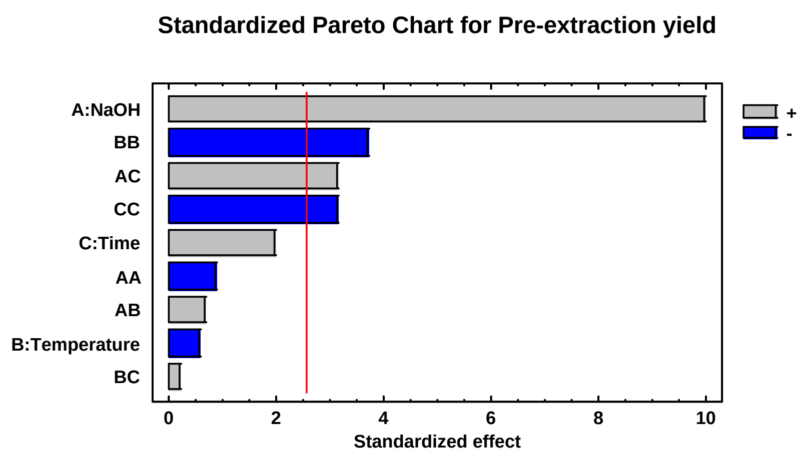


Fig. 2. Effects of process conditions on pre-extraction yield

Effects of Process Conditions on Xylan Extraction Yield

The main goal of this study was to extract some hemicelluloses from aspen chips prior to mechanical pulping. The results showed that 4 variables (NaOH charge, time, the interaction between NaOH charge and time, and the interaction between temperature and time) were the main factors affecting the xylan extraction yield, as shown in Fig. 3(a). Among these factors, NaOH charge had the most significant effect on xylan extraction yield. The xylan extraction yield increased with the increase of NaOH charge and peroxide.

Results shown in Fig. 3 are consistent with some previous work. For instance, it has been found that peroxide will promote the pre-extraction to some extent (Fang et al. 2000). Removal of hemicelluloses in a pure form from wood involves hydrolysis of ester and ether linkages between hemicelluloses and lignin. The ester bonds are easily cleaved by alkali (Sjöström 1993). The main hardwood hemicelluloses, xylan, was removed primarily by dissolution rather than by degradation, although deacetylation occurs in aqueous caustic solution (Tunc and van Heiningen 2008).

The xylan extraction yield also increased with increasing time markedly and temperature slightly, as shown in Fig. 3(b). The 3D response surface showed that xylan extraction yield increased significantly with the NaOH charge and peroxide increasing, as shown in Fig. 5(b). The maximum of xylan extraction yield was 22.55% among the conditions listed in Table 2, i.e., about 3.7% based on original wood chips.

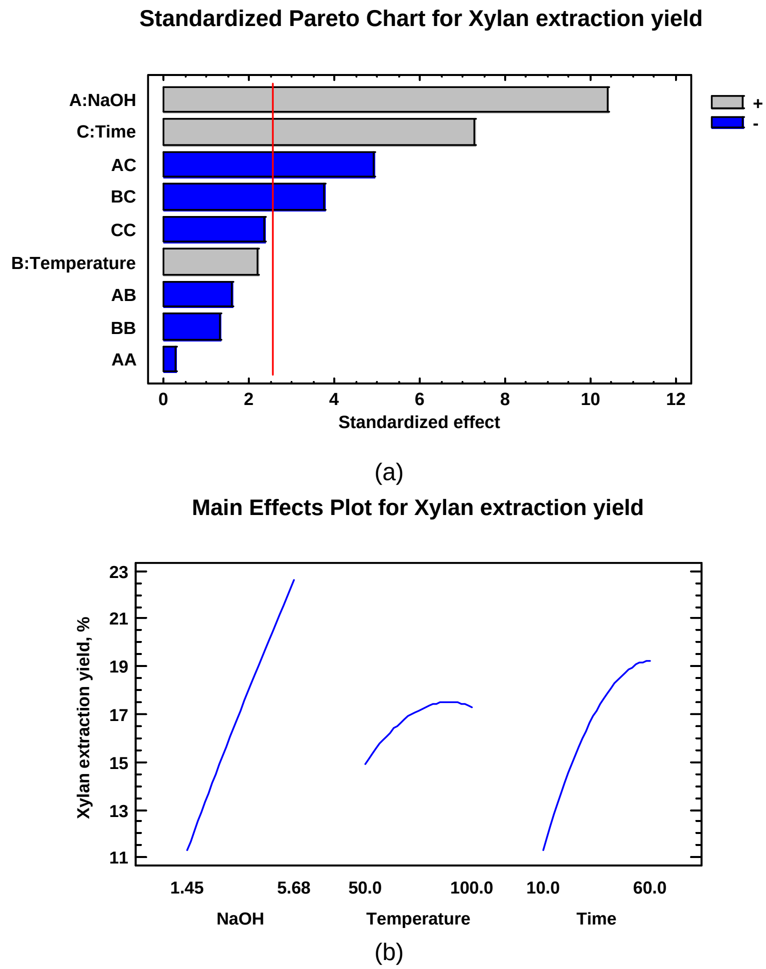
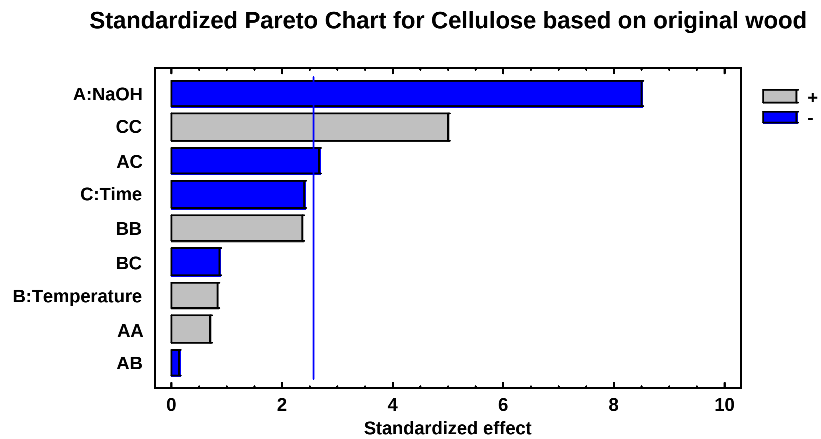


Fig. 3. Effects of process conditions on xylan extraction yield

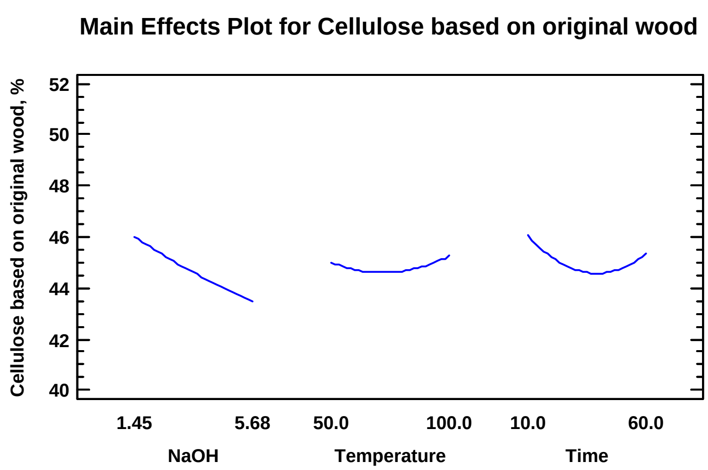
Helmerius et al (2010) extracted hemicelluloses from silver birch wood chips using either water or kraft white liquor (a mixture of NaOH, Na_2S, and Na_2CO_3 solutions). The results showed that xylan (determined as xylose) concentration was 1.1 g/L under the following conditions: L/W ratio of 3:1, 5% effective alkali (EA), 130 °C, 30 min; these results are equivalent to about 1.7% of the xylan content of the original wood. Al-Dajani and Tschirner (2007) pre-extracted hemicelluloses prior to kraft pulping from aspen chips. Their results showed that about 24.80% and 29.83% (i.e., 4.4% and 5.3% of xylan based on original wood chips) of xylan in aspen chips can be extracted under the two following conditions: (1) 50 °C, 33.3% NaOH charge, L/W ratio 4:1; (2) 90 °C, 26.7% NaOH charge, L/W ratio 4:1, respectively. Compared with these extractions conducted in the liquor phase, this study would be much more suitable for the existing chemi-mechanical pulping process. The L/W ratio in this study was so low (about 2:1) that much water will be saved; therefore the present approach results in a much higher concentration of extracted hemicelluloses streams. However, in order to minimize the pulping yield loss and degradation of chips, some hemicelluloses must be still be present in the fiber matrix. So, the optimized conditions are always a compromise between hemicelluloses extraction and the pulp properties.

Effects of Process Conditions on Cellulose Content based on Original Wood

To evaluate the effect of pre-extraction on wood chip degradation and pulping potential, the cellulose content based on original wood was analyzed. Three variables (i.e., NaOH, the interaction between NaOH charge and time, and the interaction between time and time) had the most significant effects on cellulose content based on original wood, as shown in Fig. 4. The 3D response surface illustrates that the cellulose content changed at various NaOH charges at 90 ˚C, as shown in Fig. 5(c). Figure 4(b) showed that the cellulose content based on original wood decreased with increasing of temperature and time, and then increased slightly. The minor changes were mainly due to the fact that the rates of extraction and degradation of cellulose, hemicelluloses, and lignin were different.



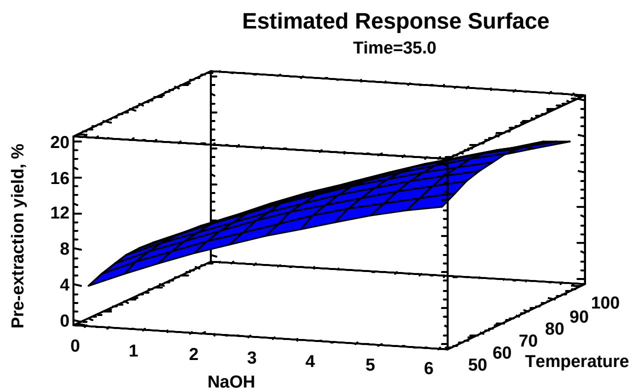
(a)



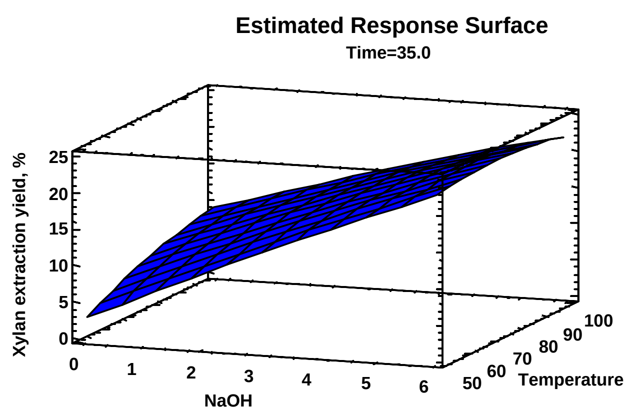
(b)

Fig. 4. Effects of process conditions on cellulose content based on original wood

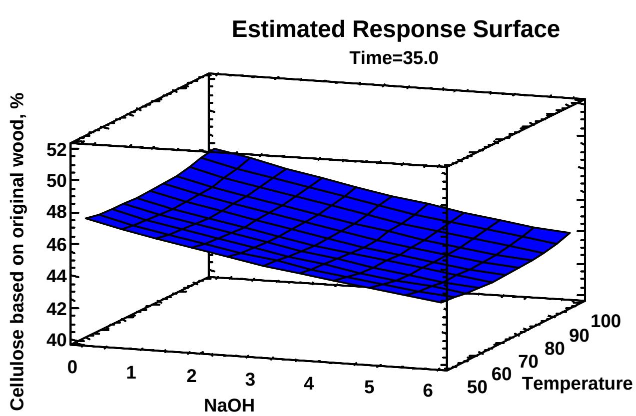
Compared with cellulose content in the original aspen chips (i.e., 47.07%), with the increase of NaOH charge, the cellulose content based on original wood chips decreased 0.17 to 3.56%. The severest degradation of cellulose (i.e., 3.56%) occurred under the following conditions: 5.68% NaOH, 75 ˚C and 60 min. Compared with the acid pre-extraction process, alkaline-based methods cause less sugar degradation (Kumar et al. 2009), which should benefit the pulping yield and pulp properties. On the other hand, cellulose cannot easily be hydrolyzed by chemicals (Taherzadeh and Karimi 2008). The hemicelluloses content (determined as xylan content) of the native aspen chips used in this study was 16.5%. The xylan extraction yield showed that 2 to 22% of xylan content was removed from the aspen chips.



(a) Pre-extraction yield



(b) Xylan extraction yield



(c) Cellulose content

Fig. 5. The estimated response surface of pre-extraction yield, xylan extraction yield and cellulose content based on original wood

Mass Distribution of Pre-extraction in M/K Digester

To investigate the mass distribution during the pre-extraction in M/K digester, a mass balance was performed on the basis of ~~100 g~~ 1 ton oven-dried aspen chips under the conditions of 5.68% NaOH, 100 °C and 35min, as shown in Fig. 6. The results indicated that about 37.3 kg of xylan could be extracted and 12.2 kg of glucan were lost from one ~~tonne~~ ton of oven-dried aspen chips under the conditions of 5.68% NaOH, 100°C, and 35 min.

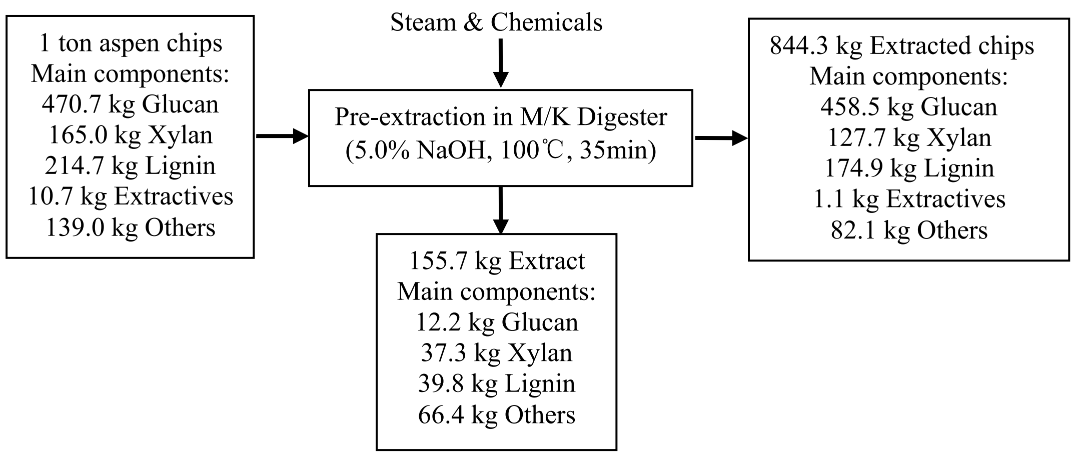


Fig. 6. Mass distribution of pre-extraction in M/K digester

CONCLUSIONS

Effects of process conditions (NaOH charge, pre-extraction temperature, duration time and their interactions) on pre-extraction yield, xylan extraction yield and cellulose content based on original wood were investigated by employing a Box-Behnken design.

The NaOH charge had the most significant effect on pre-extraction yield, xylan extraction yield, and cellulose content based on original wood. The NaOH charge, time, interactions between NaOH charge and time, and interactions between temperature and time had significant effects on xylan extraction yield. Two mathematical models of pre-extraction yield and xylan extraction yield were obtained and well verified. The maximum xylan extraction yield was 22.55%, i.e., 37.3 kg of xylan could be extracted from one ton of oven-dried aspen chips under the conditions of 5.68% NaOH, 100 °C and 35 min.

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