

Optimization of Bark Fast Pyrolysis for the Production of Phenol-Rich Bio-oil

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Bark is one of the most under-utilized types of lignocellulosic biomass in the forest industry. In this study, bark fast pyrolysis was optimized for phenols yield using response surface methodology (RSM), considering the pyrolysis temperature, gas flow rate, and biomass particle size. The bio-oil generated under optimal conditions was then characterized by gas chromatography-mass spectrometry (GC-MS), ultimate analysis, and several physical methods. A regression equation was estimated based on the statistical analysis. It was found that the optimal conditions for phenols yield were 485 °C (pyrolysis reaction temperature), 28 L/min (gas flow rate), and 0.35 mm (biomass particle size), giving an experimental phenols yield of 13.2 wt%. The bio-oil obtained in optimum conditions met ASTM standard D7544-12 and contained up to 30.42% phenols. This renewable, phenol-rich bio-oil may be a good feedstock for phenolic-based chemicals, such as phenolic resin and phenoplast.

Keywords: Lignocellulosic biomass; Bark; Fast pyrolysis; Optimization; Response surface methodology (RSM); Phenols

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INTRODUCTION

The utilization of renewable biomass to produce fuels or chemicals is now considered an essential strategy to address the issue of energy security and sustainable economics in the world. Lignocellulosic biomass is an ideal bioresource in terms of high bulk density, steady constituents, and renewable reserves. Among the parts of lignocellulosic biomass, bark is one of the most under-utilized materials in the forest industry. Currently, bark is simply landfilled or used as a low-calorie heating fuel. Generally, apart from carbohydrates and extractives, softwood bark usually contains around 40 to 55% lignin, which is a valued component with a high energy density (Pan *et al.* 2013).

Bio-oil, a multicomponent liquid mixture comprised of differently sized molecules derived primarily from depolymerization or fragmentation reactions of biomass, is convenient for storage and transportation and is highly regarded as a substituted intermediate for petroleum fuels (Czernik and Bridgwater 2004; Lu *et al.* 2009; Ren *et al.* 2013b). Fast pyrolysis of wood gives high yields of such liquids, as high as 75 wt%; these liquids can be used directly in a variety of applications or used as an efficient energy carrier (Bridgwater 2012; Mohan *et al.* 2006). Many efforts have been made to use bio-oil as combustion fuel oil or to upgrade it to transportation fuel (Bridgwater 2012; Mohan *et al.* 2006). Bio-oils have also been evaluated as a potential feedstock for fine and bulk chemicals. In particular, phenols in bio-oil have been examined for the synthesis of bio-based phenol-formaldehyde resins (Amen-Chen *et al.* 2002; Cheng *et al.* 2011; Fan *et al.* 2010).

There have been some previous reports of the conversion of the bark of different species into bio-oil. A bio-oil sample obtained by vacuum pyrolysis of softwood bark has been reported to be a valuable gas turbine fuel with a high net heating value of 32 MJ/kg and a concentration of 10 to 15% of the aqueous phase under optimal conditions (Boucher *et al.* 2000). Pine bark has also been pyrolyzed in an externally heated fixed-bed reactor, and it was found that under this slow pyrolysis condition with heating rates of 7 and 40 °C/min, oxygenated and polar fractions dominated the derived liquid (Şensöz 2003). More recently, Douglas fir and loblolly pine bark were characterized by wet chemistry methods and subjected to pyrolysis in a tubular reactor. It was found that at 500 °C, the pyrolysis of Douglas fir and loblolly pine bark generated 48.2% and 45.2% of the total oil, respectively, of which the light oils (bio-oil in the first condenser) were 14.1% and 20.7%, and the heavy oils (bio-oil condensed mainly near the end of the pyrolysis tube) were 34.1% and 24.4%, respectively (Pan *et al.* 2013). Generally speaking, previous reports have primarily dealt with the characterization of barks and investigated the yield of whole bio-oil from barks. Although many efforts have been made on this topic, efficiently and cost-effectively producing the value-added products from bark continues to be a challenge.

Larch (*Larix* spp.) is one of the most common softwood species in China, and a large amount of larch bark residues are generated every year (Ren *et al.* 2012). Currently, there is no information available on the optimal pyrolysis conditions for obtaining the highest yield of phenols from larch bark. The objective of this study was to investigate the effect of pyrolysis conditions on the specific phenols yield from larch bark and to optimize the production of the highly valued bio-oil rich in phenols by simultaneously considering the pyrolysis temperature, gas flow rate, and biomass particle size. Therefore, standard response surface methodology (RSM) design using a central composite rotatable design (CCRD) together with analysis of variance (ANOVA) was applied in this study.

EXPERIMENTAL

Materials

Larch trees were collected from the Xiao Hinggan Mountains, Heilongjiang Province, China. Larch bark generated during the debarking procedure was then used as the raw material in this study. The materials were smashed, air-dried, and screened into a series of controlled particle sizes, *i.e.*, 0.34 mm (< 0.42), 0.65 mm (0.42 to 0.841), 1.11 mm (0.841 to 1.41), 1.56 mm (1.41 to 1.68); and 1.87 mm (1.68 to 2.00). The basic characteristics, such as elemental, proximate, and chemical component compositions, were analyzed following the previously reported standard procedures (Ren *et al.* 2013c), and results are summarized in Table 1.

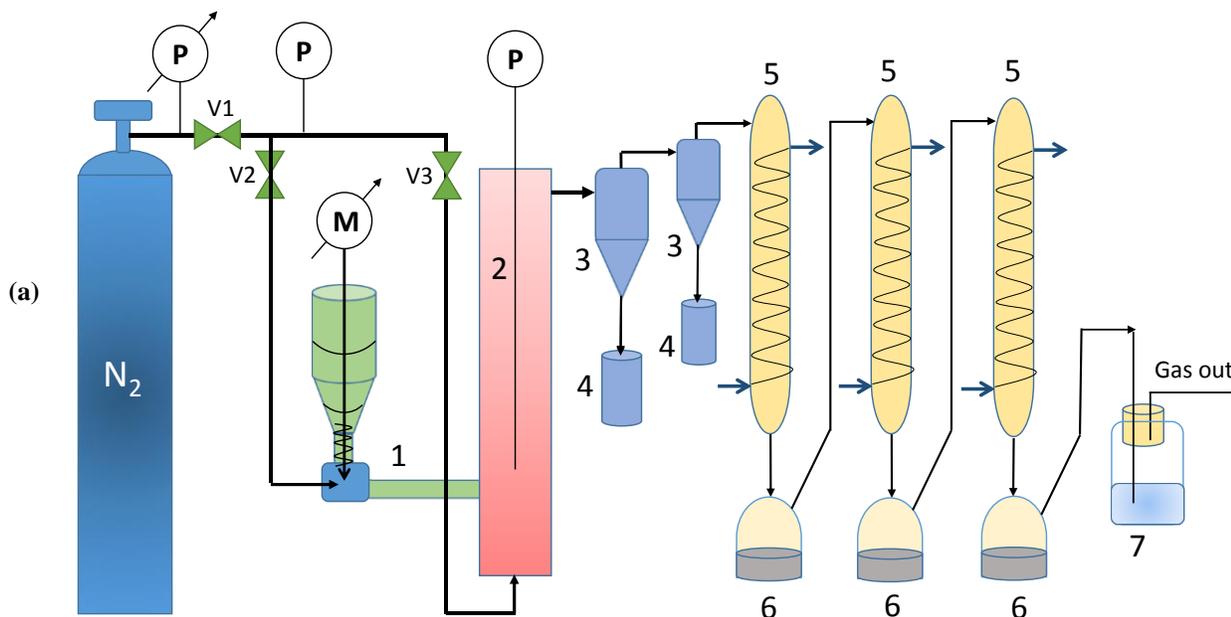
Table 1. Feedstock Analysis of Larch Bark

Elemental analysis (OD, wt%)		Proximate analysis (OD, wt%)		Chemical components (OD, wt%)	
C (%)	52.2	Volatile matter	75.3	Cellulose	28.8
H (%)	5.1	Fixed carbon	23.1	Hemicellulose	13.2
N (%)	0.2	Ash	1.6	Lignin	45.4
O (%)	42.5			Extractives	12.6

Methods

Bark fast pyrolysis in a fluidized-bed reactor system

A custom-designed pyrolysis system (shown in Fig. 1) with a nominal capacity of $1 \text{ Kg}\cdot\text{h}^{-1}$ of biomass feedstock was used to perform fast pyrolysis on larch bark to generate bio-oil.



(b)



Fig. 1. The BJFU fluidized bed pyrolysis system. (a) Schematic diagram of the system. 1, biomass feeding system; 2, fluidized bed reactor; 3, cyclone separator; 4, char collector; 5, condensers; 6, bio-oil collector; 7, gas washer. (b) Picture of the whole system

The pyrolysis unit comprised a fluidized-bed reactor, a fixed-bed reactor, and several systems for biomass feeding, char separation and collection, vapor condensation, and bio-oil recovery, all monitored with a PC. The feeding system was formed by a sealed hopper, a screw feeder of adjustable speeds, and a carrier gas tube connected to the fluidized-bed reactor. Nitrogen was used as both the carrier gas for biomass feeding and the fluidization gas in the reactor. Before being used as the fluidization gas in the main reactor, the nitrogen was preheated to around 400 °C. Silica sand was the fluidizing and heat-transfer agent in the bed.

The residence time of the biomass in the fluidized bed was estimated to be between around 1 and 2.5 s, depending on the operational gas flow rate. The rotating speed of the screw feeder, temperatures in the preheating furnace and reactors, and gas flow rates were continuously monitored and controlled by a PC. Before feeding into the reactor, all of the study materials were oven-dried overnight at 105 °C. Approximately 0.5 kg of biomass was processed per run. Bio-oil was quenched and collected by three tubular condensers in series with iced water as the cooling medium. The liquid products from these three condensers were then mixed together for utilization and analysis later. The fast pyrolysis of the larch bark occurred in the reactor in different conditions, based on the numbers given by the statistical software.

Experimental design and statistical analysis

A central composite rotatable design (CCRD) was applied to this experiment, as it can provide a specified amount of information with reduced experimental work (Abnisa *et al.* 2011). To obtain the required data, 20 experiments were performed. Pyrolysis temperature (T), particle size (S), and gas flow rate (F) were selected to study the optimization of the larch bark fast pyrolysis process, while the bio-oil yield ($Y_{bio-oil}$), water content of bio-oil (C_{water}), relative phenols content of bio-oil ($C_{phenols}$), and phenols yield ($Y_{phenols}$) were taken as the process responses. The respective value ranges of these process variables were chosen based on the literature and preliminary studies (Mohan *et al.* 2006; Mourant *et al.* 2013; Ren *et al.* 2013a). The $Y_{bio-oil}$ and $Y_{phenols}$ are specifically defined in Eqs. 1 and 2, respectively. The relative phenols content was defined as the sum of the phenols' peak area divided by the total peak area shown in the GC-MS chromatogram, which was believed to be linear with the its content in bio-oil (Lu *et al.* 2012; Ren *et al.* 2013c).

Design expert software (version 8.0.6, State-Ease, Inc., Minneapolis, MN, USA) was used to obtain the experimental matrix and the response surfaces, as well as to perform the subsequent regression analysis and analysis of variance (ANOVA). The optimum conditions with respect to phenols yield for the three variables, *i.e.*, pyrolysis temperature, gas flow rate, and particle size, were obtained using a statistical regression model and confirmed with additional experiments. The design matrices of the experiments performed, together with the results obtained, are shown in Table 2.

$$Y_{bio-oil} (wt\%) = \frac{\text{Bio-oil product (g)}}{\text{Dry bark input (g)}} \times 100\% \quad (1)$$

$$Y_{phenols} (wt\%) = Y_{bio-oil} \times (1 - C_{water}) \times C_{phenols} \quad (2)$$

Table 2. Experimental Design Matrix and Response Results of the Fast Pyrolysis of Bark

Run	Variables (studied factors)			Responses (product yields)			
	Temperature (°C)	Flow rate (L/min)	Particle size (mm)	$Y_{bio-oil}$ (wt%)	C_{water} (wt%)	$C_{phenols}$ (wt%)	$Y_{phenols}$ (wt%)
1	400.0	25.0	1.56	48.1	31.3	20.9	6.9
2	450.0	27.5	1.11	50.1	29.6	28.1	9.9
3	450.0	27.5	1.11	50.3	30.4	27.4	9.6
4	534.1	27.5	1.11	42.5	35.8	23.5	6.4
5	450.0	27.5	1.11	50.2	30.4	27.5	9.6
6	400.0	30.0	0.65	50.2	27.9	27.9	10.1
7	400.0	25.0	0.65	52.3	28.1	25.8	9.7
8	450.0	27.5	1.87	45.8	33.8	20.1	6.1
9	450.0	27.5	1.11	49.9	29.2	27.5	9.7
10	500.0	30.0	1.56	48.6	32.9	17.8	5.8
11	500.0	25.0	1.56	49.7	31.6	21.2	7.2
12	450.0	27.5	0.34	54.6	24.1	29.4	12.2
13	400.0	30.0	1.56	47.5	32.3	16.5	5.3
14	500.0	25.0	0.65	52.1	27.6	23.9	9.0
15	450.0	27.5	1.11	49.9	29.5	27.9	9.8
16	450.0	27.5	1.11	50.4	30.3	27.6	9.7
17	450.0	23.3	1.11	43.9	26.8	21.5	6.9
18	500.0	30.0	0.65	51.4	28.8	26.8	9.8
19	365.9	27.5	1.11	41.8	36.3	23.3	6.2
20	450.0	31.7	1.11	43.2	32.6	22.7	6.6

Characterization of bio-oil product

After the fast pyrolysis experiment, the bio-oil was collected in a sealed bottle and stored in a refrigerator before analyses. The water content was determined by Karl Fischer titration using CombiTitrant 5-Keto as the titrant. The pH value was measured with a pH meter at room temperature. Carbon, hydrogen, and nitrogen contents were measured using a Vario EL III elemental analyzer, and oxygen content was calculated by the difference method. The viscosity of bio-oil was measured according to ASTM D445 using a Cannon-Fenske routine viscometer at 40 °C. The higher heating value of bio-oil was acquired with a Parr 1108 oxygen combustion bomb.

Gas chromatographic-mass spectrometric (GC-MS) analyses of bio-oils were performed on a Shimadzu GC/MS-QP 2010 system. The GC separation of pyrolysis vapors was achieved with a 60 m × 0.25 mm × 0.25 μm DM-5 column. Helium was used as the carrier gas with a constant flow rate of 1 mL/min. The injector split ratio was fixed at 50:1. The injector and detector temperatures were set at 250 and 280 °C, respectively. The GC oven temperature was programmed to start at 40 °C for 5 min and then to rise to 280 °C at the heating rate of 5 °C/min. The mass spectrometer was operated in EI mode at 70 eV, and the mass spectra were obtained from m/z 20 to 400. Before sampling the bio-oil for analysis, it was taken from the refrigerator and held at room temperature for 1 h to allow it warm up. Then it was mixed for 5 min by a vortex shaker. The bio-oil sample was prepared as a 5 wt% solution dissolved in acetone filtered through a 0.25-μm PTFE filter. One microliter of bio-oil solution was injected for each analysis. Peak identification was completed using the NIST mass spectrum library and data from previous studies (Bahng *et al.* 2009; Kim *et al.* 2011; Mendu *et al.* 2011; Ren *et al.* 2013b).

RESULTS AND DISCUSSION

Effect of Fast Pyrolysis Conditions on the Phenols Yield from Larch Bark

The three-dimensional (3-D) response surfaces for the optimum yield of phenols are shown in Fig. 2.

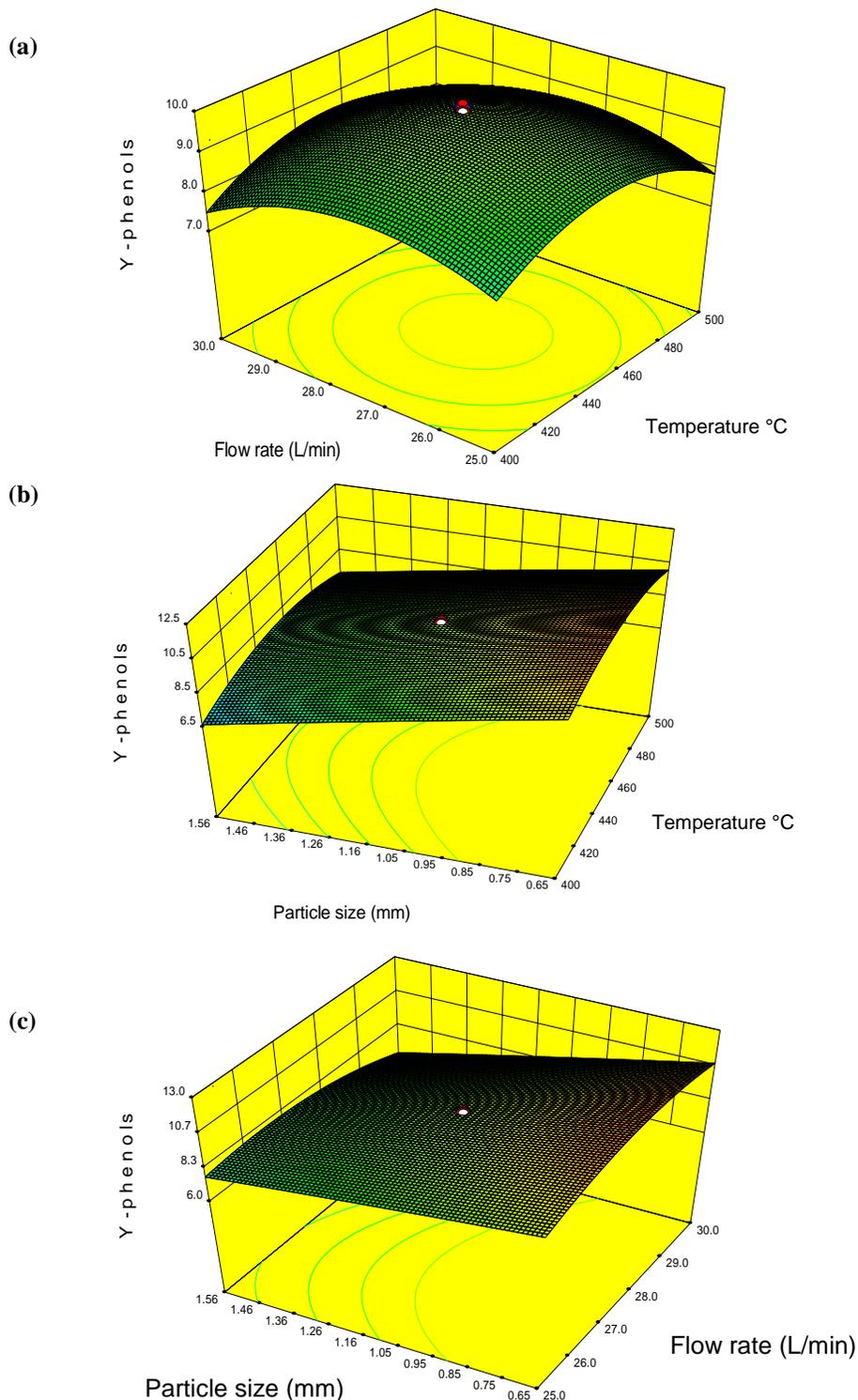


Fig. 2. The 3-D response surface of the phenols yield from the fast pyrolysis of larch bark: (a) the effect of gas flow rate and pyrolysis temperature; (b) the effect of biomass particle size and pyrolysis temperature; (c) the effect of biomass particle size and gas flow rate

Parts 2a, b, and c of the figure depict the interaction effects of the gas flow rate, pyrolysis temperature, and biomass particle size in pairs with 3-D response surface plots. According to the trends shown, these three parameters affect the phenols yield in different ways during the fast pyrolysis process.

The phenols yield increased with decreasing particle size, which can be explained by the enhanced degradation of the lignin in biomass. With smaller particle size, biomass particles experienced higher heating rate inside of the pyrolysis reactor, which favored the further cracking of the lignin fraction, which is typically hard to completely decompose within a short time. The effects of biomass particle size (0.18 to 5.6 mm) on the yield and composition of bio-oil from the pyrolysis of Australian oil mallee woody biomass were investigated in a fluidized bed reactor at 500 °C (Shen *et al.* 2009), and it was found that the yield of bio-oil decreased as the average biomass particle size was increased from 0.3 to about 1.5 mm. The authors ascribed this result to the impact of particle size in the production of lignin-derived compounds.

In contrast to the linear effect of biomass particle size, pyrolysis temperature and gas flow rate had a parabolic effect on the phenols yield. With increasing pyrolysis temperature and gas flow rate, the phenols yield gradually increased to the highest value at moderate pyrolysis conditions and then decreased when the temperature and flow rate increased further. The highest yield of phenols was observed at around 450 °C. The results here are comparable to those reported for the bio-oil yield from mallee bark (Mourant *et al.* 2013).

The gas flow rate primarily affects the residence time and heat transfer of biomass particles inside the reactor. In general, slower gas flow rates reduce the efficiency of heat transfer experienced by biomass, while higher gas flow rates decrease the residence time of particles and then may let the biomass feedstock pass through the fluidized bed reactor without complete degradation.

Development of Regression Model Equation and Statistical Analysis

The quadratic model for the phenols yield from larch bark fast pyrolysis, as determined by Expert Design software, is represented as Eq. 3, where the target dependent phenols yield ($Y_{phenols}$) is given as a function of biomass particle size (S), gas flow rate (F), and temperature (T). Only terms determined to be statistically significant ($p = 0.05$) were included in the prediction equation. The positive and negative signs of the independent variable coefficients indicate synergistic and antagonistic effects of the parameter in the equation, respectively. The quality of the models was judged from their coefficients of correlation and the comparison between the actual and predicted values. Regression analysis showed that the coefficient of determination was satisfactory for the prediction of the phenols yield from larch bark fast pyrolysis. Additionally, the relationship between the actual and predicted value of the relative yields of phenols for all 20 tests (different gradient color means the different location or value range of the phenols yield) by regression analysis is given in Fig. 3. The data points are all located very close to the diagonal line, which indicates an acceptable fit of the developed quadratic model for the experimental results.

$$Y_{phenols} = 9.11 + 4.83S - 0.31FS - (1.06 \times 10^{-6}) T^2 + (4.79 \times 10^{-3}) F^2 \quad (3)$$

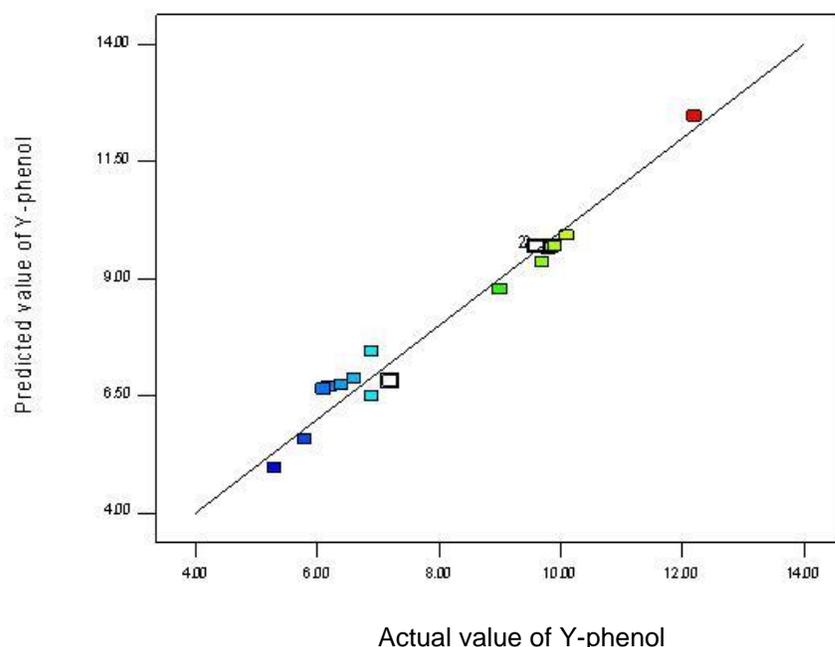


Fig. 3. The relationship between the actual and predicted values of the relative yields of phenols by regression analysis ($R^2 = 0.9617$, Adj $R^2 = 0.9515$)

To explore the correlation and further confirm the adaptability of the model, a summary of the analysis of variance (ANOVA) for the model of phenols yield is shown in Table 3.

Table 3. Analysis of Variance (ANOVA) of the Response Surface Quadratic Model for Phenols Yield of Larch Bark Fast Pyrolysis

Source	Sum of squares	Degree of freedom	Mean of square	F-value	P-value Prob>F
Model	69.52	9	7.73	41.61	< 0.0001
T- Temperature	0.0014	1	0.0014	0.0073	0.9335
F- Gas flow rate	0.39	1	0.39	2.10	0.1785
S- Particle size	40.99	1	40.99	220.73	< 0.0001
TF	0.045	1	0.045	0.24	0.6331
TS	0.41	1	0.41	2.18	0.1705
FS	2.21	1	2.21	11.88	0.0063
T ²	16.10	1	16.10	86.71	< 0.0001
F ²	11.62	1	11.62	62.57	< 0.0001
S ²	0.035	1	0.035	0.19	0.6729
Residual	1.86	10	0.19	—	—
Lack of Fit	1.79	5	0.16	1.17	0.4142
Pure Error	0.068	5	0.014	—	—
Cor Total	71.38	19	—	—	—

The ANOVA resulted in an F value of 41.61 and a P value lower than 0.0001, showing that the model was highly statistically significant. Moreover, the lack of fit F value was 0.4142, which means that the lack of fit is not significant in relation to the pure

error. Therefore, particle size (S), the interaction effects between the gas flow rate and particle size (FS), the squared effect of the pyrolysis temperature (T^2), and the squared effect of the gas flow rate (F^2) were significant model terms with regard to phenols yield and were thus selected as the independent variables for the developed regression model (Equation 3). The regression model was successfully developed by response surface methodology and also confirmed by the statistical tool. It will be a practical tool in the biomass thermo-chemical conversion area, which can be used not only to optimize the phenols production from the fast pyrolysis of bark, but also to help better understand the effect of process conditions on the target product.

Characterization of Bio-oil Produced in Optimized Conditions

To maximize the phenols yield, the DOE (design of expert) model suggested the optimal operating conditions were a pyrolysis temperature of 485 °C, a gas flow rate of 29 L/min, and a biomass particle size of 0.35 mm. The experimental results and the value predicted by the regression model are presented in Table 4. The predicted phenols yield at optimal conditions was as high as 13.4 wt%. An experimental value of 13.1 wt% with a standard deviation of 0.2 was obtained, indicating that the data calculated by the model and the experimental data fit well.

Table 4. Results of the Model Evaluation

Fast pyrolysis conditions			Phenols yield (wt%)	
Temperature (°C)	Flow rate (L/min)	Particle size (mm)	Predicted	Experimental
485	28	0.35	13.4	13.2 (0.2)

The water content, pH, viscosity, calorific value, and ultimate composition of the bio-oil generated under optimal conditions are summarized in Table 5. The physical properties of bio-oil obtained here met the specifications listed in ASTM D7544-12 (ASTM 2012). The water in bio-oil resulted from dehydration reactions during fast pyrolysis, as well as some bound water inside the biomass. The presence of water in bio-oil improved its flowability but also resulted in a lower heating value and a higher tendency for phase separation. The acidity of bio-oil is attributed to the presence of acidic compounds such as acetic acid and formic acid, primarily resulting from the degradation of carbohydrates in the feedstock. Bio-oil from larch bark has a higher heating value of 19.6 MJ/kg, which is around half that of No. 4 fuel oil (Tzanetakis *et al.* 2011). Because bark has a higher carbon content than wood, the carbon content of the derived bio-oil from larch bark was also slightly higher than the bio-oil from wood (Kim *et al.* 2013).

Table 5. Properties of Bio-oil Produced in Optimized Conditions

Elemental analysis (wt%)		Physical properties	
C (%)	51.2	Water content (wt%)	25.2
H (%)	6.1	pH	3.11
N (%)	0.4	Viscosity at 40 °C	34.7
O (%)	42.3	Higher heating value (MJ/kg)	19.6

The bio-oil from larch bark was primarily composed of oxygenated organic compounds, such as alcohols, ketones, aldehydes, acids, furans, esters, ethers, sugars, phenols, and nitrogenous compounds (Fig. 4). More than 60 peaks appeared on the chromatogram, and around 40 of them were identified by comparing the mass spectra of each peak with those of authentic compounds in the National Institute of Standards and Technology (NIST) mass spectra library, then semi-quantified by recording the peak areas of the compounds. Ketones, acids, furans, sugars, and phenols were the main categories equal to or more than 10% in the bio-oil from larch bark. It has been reported that cellulose and xylan mainly release sugars and ketones, respectively, while phenols originate from the degradation of lignin (Ren *et al.* 2013b). Particularly, the relative content of phenols in the bio-oil from bark in this work was 30.42%, much higher than bio-oils from other types of biomass, such as wheat straw, switchgrass, *Miscanthus*, and beech wood (Greenhalf *et al.* 2013). The high content of phenols in bio-oil from larch bark indicated its potential as a renewable substitute for aromatics. More specifically, this bio-oil may be a useful material to produce phenolic-based chemicals, such as phenolic resin and phenoplast.

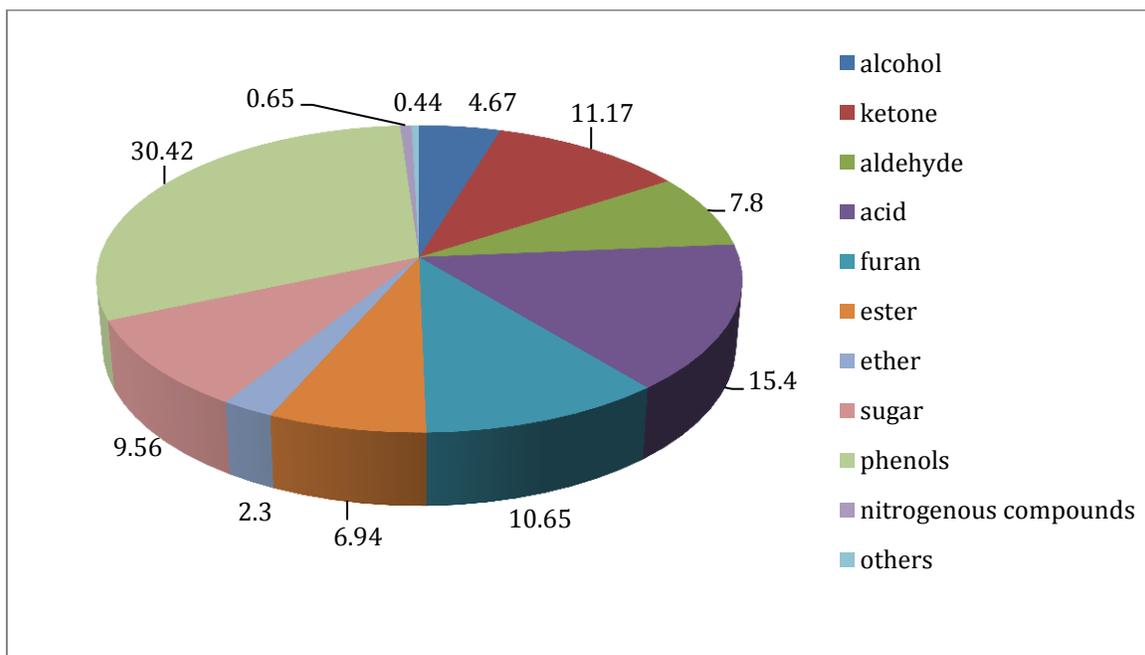


Fig. 4. Chemical composition of bark bio-oil obtained in optimized conditions

CONCLUSIONS

1. It was found that the phenols yield increased as the particle size of the biomass decreased. Pyrolysis temperature and gas flow rate showed a parabolic effect on the yield of phenols, and higher phenols yield was obtained at a moderate temperature and gas flow rate.
2. A regression equation was estimated based on the statistical analysis, and the optimized conditions for phenols yield were 485 °C (pyrolysis reaction temperature), 28 L/min (gas flow rate), and 0.35 mm (biomass particle size), giving an experimental phenols yield of 13.2 wt%.

3. Larch bark bio-oil generated in optimal conditions was characterized by GC-MS, ultimate analysis, and several physical methods. It was found that the bio-oil met the ASTM standard D7544-12, and its high content of phenols (30.42%) indicated its potential as a renewable substitute for phenolic-based chemicals.

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