

INFLUENCE OF FAST PYROLYSIS CONDITIONS ON POLYPHENOLS FRACTION FROM BIO-OIL

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ABSTRACT

The aim of this work was to develop a pyrolysis process for polyphenols fraction bio-oil, produced by fast pyrolysis of *Larix gmelinii* circulated-fluidized bed (SCFB). This paper is focused on the process examination and product analysis. A range of operating parameters, including temperature (T) from 500°C to 600°C in the SCFB, and particle size (d) from 0.2-0.3 mm to 0.9-1.2 mm, and feed rate (n) from 20 r • min⁻¹ to 50 r • min⁻¹, and gas flow (Q) from 15 m³•h⁻¹ to 30 m³•h⁻¹ in the SCFB was conducted to the effect on the content of phenolic compounds (COP) bio-oil derived from *Larix gmelinii*. It was determined that the order of the parameters to influence on the COP in bio-oil was T > d > n > Q. The temperature and particle size of the parameters played key roles in the process. The operating parameters for increasing the COP of bio-oil were determined, i.e. temperature 550°C, particle size 0.3-0.45 mm, feed rate 50 r • min⁻¹ and gas flow 15 m³ • h⁻¹.

KEYWORDS: Woody biomass, pyrolysis, circulated-fluidized bed (SCFB), compounds.

INTRODUCTION

As a renewable, plentiful and environmental friendly resource, biomass is considered to be a promising energy material. Developing and utilizing biomass resources will positively contribute

to the sustainable development goal for the whole world.

Producing bio-oil with high calorific value by fast pyrolysis of wood residues has been studied extensively the recent years. Yang et al. (2005) studied the effect of reaction temperature on the products of wheat straw from fast pyrolysis on the spouted-fluidized bed. Du et al. (2007) analyzed the factors affecting yield of bio-oil from fast pyrolysis of wood biomass. Yang et al. (2008) produce bio-oil with fir sawdust as the raw material using the vacuum pyrolysis technology. studies producing bio-oil with vacuum pyrolysis were completed by Murwanashyaka et al. 2001a, Boucher et al. 2000a, b, Murwanashyaka et al. 200b, Garcia-Perez et al. 2002, 2007. Liu et al. (2010) effect of reaction conditions on the yield of bio-oil. He et al. (2010) higher yield of liquid product through the direct catalysis experiment on fluidized bed pyrolysis fixed-bed reactor. Zhu et al. (2007) described the optimized self-heated pyrolysis process combined with three kinds of developed reactors.

But fewer work was reported on the influence factor of targeted compounds in pyrolysis product. Therefore, it is necessary to perform further study on this issue and acquire the optimized pyrolysis condition.

In study, *Larix gmelinii* bark (from Daxinganling District in north east of China) was pyrolyzed on spouted- circulated- fluidized bed (SCFB) which was designed by Beijing Forestry University. The properties of bio-oil were characterized to explore the impact of react condition on the phenolic compounds content of bio-oil in order to increase the phenolic content of bio-oil by optimizing fast pyrolysis process.

MATERIAL AND METHODS

Equipments and methods

GC-MS analysis method

GC-MS (TRACE GC-VOYAGER, America ThermoQuest company) was used to analyze the content of phenolic compounds in bio-oil. The compounds and contents of bio-oil were determined by a GC-MS system (TRACE GC-VOYAGER, Shimadzu, Columbia, MD, USA) equipped with a 60 × 0.32 mm column with a 0.5 μm Film-Thickness. Then, the content for each compound was calculated by Internal Reference Method.

MS: ionization mode EI, electron bombardment energy 70 ev, charging multiplier tube voltage 500 V, scanning range was from 50 to 500 u and scanning time was 1 s.

The temperature program was starting from 40°C for 3 min, then increasing at 6°C. min⁻¹ till 270°C and maintained for 5 min.

Fast pyrolysis method

SCFB fast pyrolysis based on fluidized bed reactor with the recirculating of non-condensable gas as flow carrier (in). It up of reactor (1), feeder (2), (15), system(16), system (5, 6, 7 and 8) and a separator (3 and 4).

Fast pyrolysis was performed as following procedure in SCFB system. Passing the rotameter, the small part of the fluidized gas was injected into the bunker to balance the pressure, and most of it was preheated and injected into SCFB reactor to fluidize the ceramic ball grain which were heated. At the same time, the *Larix gmelinii* bark was fed into the reactor by a screw-feeder for touching the ceramic ball and was cracked into the pyrolysis vapors and the char. Then the vapors and char were entered into the cyclone separator to be separated. Char was collected in char collector, while the vapors went through condensers (5-8) be condensated into liquid and gas.

liquid was bio-oil and the gas was noncondensable gas. The oil was collected into liquid collecting bottle (9-12). The uncondensable gas was entered into the filter, to remove the residual liquid and tiny char, one part involved in the circulation, the rest was exhausted after measurement by rotameter (Fig. 1).

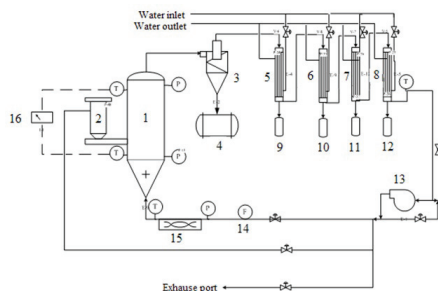


Fig. 1: Flow chart for SCFB fast pyrolysis system. 1- Spouting-circulating fluidized bed reactor: 2 - Screw-feeder: 3- Cyclone separator: 4- Char collector: 5- Condenser I :6 - Condenser II: 7 - Condenser III: 8- Condenser IV: 9 - Liquid collecting bottle I: 10 - Liquid collecting II: 11 - Liquid collecting III: 12 - Liquid collecting IV: 13- Roots blower: 14 - Flowmeter: 15 - Heater: 16 - Monitoring system.

Materials

Raw material preparation: Larch bark was crushed in the grinder, and screened for four particle sizes by standard sieve respectively: 0.2-0.3, 0.3-0.45, 0.45-0.9 and 0.9-1.2 mm. mcontent were 10.3 %.

Silica gel: Adsorption material, analytically pure. After activated for four hours in the drying oven (105°C), then silica gel was put into dryer beused.

Other reagents: tetrahydrofuran, cyclohexane, toluene and ethanol, all were analytically pure.

Column chromatography of the bio-oil

The bio-oil separated fractions by column chromatography, and each with GC-MS.

Before column chromatography, the bio-oil sample should be pre-treated , the carbon residue was filtered from the sample by the soxhlet extractor with 2 layer filter paper at vaccum degree 0.08 MPa. Then, the water was separated from the filtered sample with the rotary evaporator at 50°C and degree 0.08 MPa. Tchar and the water bio-oil were separated to improve the effect of column chromatography.

Activated silica gels (particle size 0.076 ~ 0.150 mm) were cooled to 80°C in the dryer, and then packed quickly in a glass tube (with the inner-diameter of 30 mm and fiber glass for support in the one end) with the height of 0 mm, and distributed uniformly and densely in the glass tube. At the same time, the glass tube whends were sealed with cyclohexane while the silica gels in it was wetted with cyclohexane immediately. Certain amount of pre-treated bio-oil (pre-treated bio-oil and silica mass ratio of 1:35) was dropping into the glass tube end with no-fiberglass. After 5 minutes, three solvents of cyclohexane, toluene, ethanol were used for elution in sequence, three fractions were separated successively with different elution solvents, which are: Cyclohexane eluted fractions, toluene elution fractions and ethanol eluted fractions. There negative pressure of - 0.01 MPa to accelerate the eluting speed at the bottom of the glass tube. The solvent in the eluting solution firstly distilled away, and then the rest bottom solution phenolic compounds in the bio-oil by analyzing all fractions by GC-MS.

Design of experiment

In order to study the fast pyrolysis process for increasing COP in bio-oil, the orthogonal design test was used. According to orthogonal design theory, factors and 4 levels (Tab. 1) were selected to the effect of reaction temperature (T), larch bark particle size (d), the feed rate (n) and gas flow (Q) COP in bio-oil factors and levels of orthogonal were shown in table . The testing program was described in Tab. 2 in which B column was designated blank column for the ror evaluation.

Tab. 1: and levels design.

Factor	T(°C)	D(mm)	n(r•min ⁻¹)	Q(m ³ •h ⁻¹)
1	500	0.45-0.9	20	15
2	500	0.3-0.45	30	20
3	550	0.2-0.3	40	25
4	600	0.9-1.2	50	30

Tab. 2: Design parameters for larch bark pyrolysis.

Condition	T(°C)	d (mm)	n (r•min ⁻¹)	Q (m ³ •h ⁻¹)	B	COP (%)
1	500	0.45-0.90	20	15	1	20.12
2	500	0.30-0.45	30	20	2	27.69
3	500	0.20-0.30	40	25	3	11.32
4	500	0.90-1.20	50	30	4	17.33
5	500	0.45-0.90	30	25	4	17.62
6	500	0.30-0.45	20	30	3	23.70
7	500	0.20-0.30	50	15	2	31.67
8	500	0.90-1.20	40	20	1	9.50
9	550	0.45-0.90	40	30	2	30.97
10	550	0.30-0.45	50	25	1	35.02
11	550	0.20-0.30	20	20	4	30.12
12	550	0.90-1.20	30	15	3	27.11
13	600	0.45-0.90	50	20	3	28.73
14	600	0.30-0.45	40	15	4	28.33
15	600	0.20-0.30	30	30	1	27.23
16	600	0.90.2	20	25	2	14.21

Selection of reaction temperature

The biomass in the pyrolysis reactors an interaction the temperature rise of the biomass and pyrolysis of the biomass at the same time. The final pyrolysis product of biomass include gas, carbon and bio-oil, which depended on the reaction temperature. When pyrolysis temperatures low, pyrolysis main products char. However, with the increases in temperature, biomass be more fully pyrolyzed, further producing more bio-oil, but if the temperature exceed °C, be translated to non-condensated gas, leading to decrease in bio-oil content. On the other hand, two primary pyrolysis reaction ha produced biooil further cracking or polymerization, then translating into non-condensable gas, carbon or other small molecules and molecules of liquid products. Large amounts of literature indicated that at temperature of °C (Islam and Zailani 1999, Hoirne and Williams 1996, Onay and Kockar 2003, Yu et al. 1997, Thangalazhy et al. 2011), bio-oil ha the largest yield and phenolics content of oil reach the aximum. Therefore, in this work fast pyrolysis done at 500 °C, 550 °C, and 600 °C.

Selection of larch bark particle size

Biomass grain size heating rate and devolatilization rate of, so that the behavior of biomass pyrolysis s changed too big size face had higher temperature than its centre when it was pyrolyzed. The bigger size of grain, the temperature difference was more significant. From the face to the centre, the grain temperature grade was not consistent. The temperature grade in grain face was higher than its centre. That is when the face of grain was pyrolyzed sufficiently, the centre of grain could not be fully pyrolyzed. There was a slower heating ratio in the centre than the face of the too big size grain so that the yield of bio-oil from the centre was lower than the face of grain. The time that the pyrolysis vapour from the centre of grain went to the face of big size grain was longer than small size. The pyrolysis vapour could be further cracked or polymerized going from the centre to the face of grain, making the increase in charcoal and gas yield, and the decrease in bio-oil yield. Too small size of grain could increase bio-oil yield also increased the cost of producing the grain from biomass. A large number of studies show that the biomass particle size at about 1mm s beneficial to the yield of bio-oil when taking into account the cost (Beis et al. 2002, Encinar et al. 1998, Li et al. 2004, Westerhof et al. 2010). Therefore, in this work, bark particle sizes selected with 0.2-0.3, 0.3-0.45, 0.45-0.9, and 0.9-1.2 mm.

Selection of the feed rate

The feeds selected were 20, 30, 40, and 50 $\text{r}\cdot\text{min}^{-1}$ according to technical requirements of spiral feeder speed in the fast pyrolysis system.

Selection of gas flow

The process of biomass pyrolysis is affected by many kinds of factors. When the enter into the pyrolysis reactor, the temperature was increased to reach the pyrolysis reactor, and the state process of temperature rising need certain time. The more time the was heated the higher temperature it had. The pyrolysis process of biomass ha the deeper finish. At the same time, biomass c be decomposed into ondensing gas, non-condensable gases and carbon due to the chemical bonds fracture. Condensing gas react more with hot carbon and bio-oil when they contacted each other, and part of non-condensable gases s generated. Reasonable retention time of the pyrolysis vapours (that is compound of the condensing gases and noncondensable gases) s very important for biomass full transformation. In order to maximize their bio-oil production, retention time of pyrolysis vapours should be shorten, making the pyrolysis to leave quickly from reactor, and reducing bio-oil second crack; on the other hand, in order to obtain high carbon output, retention time of pyrolysis vapours should try to be extended and remained volatile product in the reactor. flow influenced on the retention of the pyrolysis vapours. The increasing the gas flow could decrease the retention time of the pyrolysis vapours. In general, high temperature pyrolysis of biomass having time of 1 second s good for the improvement increase of bio-oil yield (Caglar and Demirbas 2002, Zansi et al. 2002). In this work, flow was selected at 15, 20, 25, and 30 $\text{m}^3 \cdot \text{h}^{-1}$.

RESULTS AND DISCUSSION

Analysis of range

The range analysis was aimed to clarify the significance levels of different influencing factors on the content of phenolic compounds. In addition, those most significant factors could be disclosed based on the result of range analysis.

Tab. 3 summarizes the statistics analysis of the effect of different factors on the content of

phenolic compounds value for each level of a parameter was the sum of four values shown in Tab. 3, and the optimal level of variables can be obtained by comparing with mean(M) value. The range value (R) for each factor was the difference between the maximal and minimal value of the four levels and R scales the effect of variables on the response. High R of variable mean that this variable has strong effect on the response. The rang for B column was the smallest in five rangs in Tab. 3, that meant the four factors were selected rightly for influence on the content of phenolic compound in bio-oil. The rang for T column was the highest in the five rangs declared T was the primary factor in the selected factors. Based on the results of range analysis, the significance sequence of all the investigated factors was lined. The influence order of each factor was $T > d > n > Q$.

As the analysis of range can not be the error during the experiment and the results, the analysis of variance was processed under different conditions.

Analysis of variance

In our experiments, a blank column was set in the orthogonal Table for error estimate, thus, it was unnecessary to repeat the experiments for each protocol. The sum of squares of deviation, degree of freedom, and squared deviation of reaction temperatures, particle sizes, feed rate and gas flows were determined and summarized in Tab. 4.

As show in Tab. 4, the reaction temperature and particle size had shown significant influences on the content of phenolic compounds due to its 'F' being larger than ' $F_{1-0.05}$ ', less than ' $F_{1-0.01}$ ', and feed rate and gas flow had little effect on the content of phenolic compounds due to its 'F' being less than ' $F_{1-0.05}$ '. So in order to improve the content of phenolic compounds, reaction temperature and particle size should be focused on.

Tab. 3: Ranges for various factors.

Factor	T	D	N	Q	B
M1	19.12	25.0	22.04	26.81	22.97
M2	20.62	28.69	24.91	24.01	26.14
M3	30.81	24.36	20.03	19.54	22.72
M4	24.63	17.04	28.19	24.81	23.35
R	11.69	11.65	8.16	7.27	3.42

Tab. 4: Significance for various factors.

Resources of variance	Square sum	Degree of freedom	Mean square	F	Significant
T	327.18	3	109.0	10.87	*
d	286.24	3	95.41	9.51	*
n	151.23	3	50.41	5.02	
Q	112.92	3	37.64	3.75	
Error	30.10	3	10.03		

Note: $F_{1-0.01}=5.5$ $F_{1-0.05}=2.8$. **Most significant difference, *Significant difference, Non-significant difference in blank.

Intuitive analysis

The effect of reaction temperature on content of phenolic compounds

Fig. 2 showed that between 500°C and 550°C, the content of phenolic compounds increased significantly with the temperature rose, and reached a maximum at 550°C, after that the content

of phenolic compounds was significantly decreased. It indicates that during the fast pyrolysis process, the lignin was easy to produce phenolic compounds as the temperature increases between 500 and 550°C. Main reason for the trend mentioned above could be the group on side chain of lignin and the methoxy on aromatic ring easy to be fractured to produce primary products—the guaiacol-type and syringol-type compounds, the temperature was above 550°C, these two products split into volatile component (CO, CO₂ and CH₄ etc.), which reduce the content of phenolic compounds (Shen et al. 2010).

The effect of particle size on content of phenolic compounds

Fig. 2 showed that the particle size was between 0.2 and 0.45 mm, the content of phenolic compounds increased as the particle size increased, while when the size was greater than 0.45 mm, the content decreased sharply as the size increased. It is because the heat conduction has little effect on fast pyrolysis process, however, the mass transfer has great effect when the particle size is between 0.2 and 0.45 mm. Increasing of the particle size results in the difficulty of the split of gas, then intensifies the second reaction of gas, which could increase the content of phenolic compounds. The particle size increase 0.45 mm or more, fast pyrolysis is affected mainly by heat conduction, the excessive increased particle size reduced the temperature internal of the particle and reduced the yield of phenolic compounds.

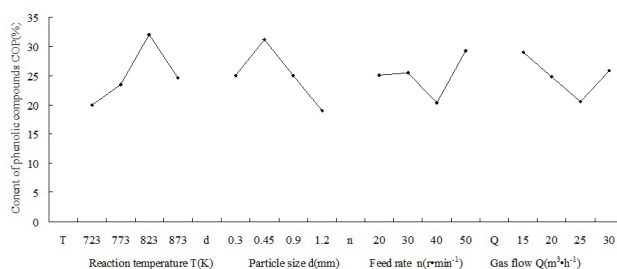


Fig. 2: Relationship between phenol content in bio-oil and various factors and level.

The effect of feed rate on content of phenolic compounds

Fig. 2 showed that the feed rate was between 20 and 30 r·min⁻¹, the content of phenolic compounds was no change, while when the feed rate was between 30 and 40 r·min⁻¹, as the feed rate increased, the content of phenolic compounds decreased significantly, and reached the minimum when the feed rate was 40 r·min⁻¹, then became increased. This was due to differences of the volume of reactor, when the feed rate between 20 and 30 r·min⁻¹, the feed rate was insufficient compared to the volume which resulted in the great change on the gas residence time in the reactor; when the feed rate was 40 r·min⁻¹, due to the feed rate and gas residence time became reduced, the probability for the second reaction was reduced, which reduced the yield of phenolic compounds. When the feed rate continued to 50 r·min⁻¹, the materials in reactor increased within unit time, and the gas could not be from the surface of material, increasing the gas residence time and the second reaction, and the yield of phenolic compounds increased (Wang et al. 2009). But it indicates in the analysis of variance, compared to the reaction temperature and particle size, within the range of feed rate, the feed rate has little effect on the content of phenolic compounds.

The effect of gas flow on content of phenolic compounds

It can be seen from Fig. 2, the content of phenolic compounds in bio-oil decreased significantly as the increase of gas flow, reached the minimum when the gas flow was 25 m³·h⁻¹,

and then increased significantly was mainly due to increasing of the gas flow, the gas concentration came from the larch bark interface between two-phase (gas and solid) decreased, the gas can break from the surface of particle immediately, meantime, the residence time of the gas that produced during the process decreased (Wang et al. 2009), the chance for second reaction decreased, resulted in the lower yields of phenolic compounds; when the gas flow came to over $25 \text{ m}^3 \cdot \text{h}^{-1}$, the fluidization of materials in SCFB increased, which improved the heat transfer, result in deep pyrolysis of biomass, thereby increase the production of phenolic compounds. However, analysis of variance indicate that, compared to the reaction temperature and particle size, within the range of gas flow selected in experiment, the gas flow has little effect on the content of phenolic compounds.

CONCLUSIONS

In the experimental range, reaction temperature and particle size had significant effect on the content of phenolic compounds, however, the feed rate and gas flow had non-significant effect.

The content of the phenolic compounds was the highest at the 550°C of the four temperature levels, indicated the temperature of 550°C was selected for optimized temperature level in the selected temperature levels for the content of phenolic compounds. The content of the phenolic compounds was the highest at the 0.3-0.45 mm of the four particle size levels seen

Fig. 2, indicated the particle size of 0.3-0.45 mm was selected for optimized particle size level in the selected particle size levels for the content of phenolic compounds. The content of the phenolic compounds was the highest at the $50 \text{ r} \cdot \text{min}^{-1}$ of the four feed rate levels seen Fig. 2, indicated the feed rate of $\text{r} \cdot \text{min}^{-1}$ was selected for optimized feed rate level in the selected feed rate levels for the content of phenolic compounds. The content of the phenolic compounds was the highest at the $\text{m}^3 \cdot \text{h}^{-1}$ of the four gas flow levels seen Fig. 2, indicated the gas flow of $\text{m}^3 \cdot \text{h}^{-1}$ was selected for optimized gas flow level in the selected gas flow levels for the content of phenolic compounds. Therefore, highest content of phenolic compounds came out under the process that: $T = 550^\circ\text{C}$, $d = 0.3\text{-}0.45 \text{ mm}$, $n = 50 \text{ r} \cdot \text{min}^{-1}$, $Q = 15 \text{ m}^3 \cdot \text{h}^{-1}$. The content of phenolic compounds was 37.2 % under the optimized process.

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