

ORIGINAL RESEARCH ARTICLE

Analysis of chemical components and liquefaction process of eucalyptus globulus bark

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ABSTRACT

For 5-year-old *Eucalyptus globulus*, the optimal liquefaction bark process was explored by analyzing the chemical components of the bark and its liquefaction residue before and after the liquefaction. The results of chemical component determination showed that the contents of cellulose, hemicellulose and lignin of bark were 36.65%, 18.98%, and 45.37% respectively. The contents of benzene-alcohol extractives, hot-water extractives and 1% NaOH extractives were 10.30%, 7.15%, and 23.64% respectively. The content of ash accounted for 7.49%. The liquefaction process showed that the catalytic effect of concentrated sulfuric acid was better than concentrated phosphoric acid. The optimum liquefaction parameters were catalytic of 3% concentrated sulfuric acid, temperature of 160 °C and the liquid-solid ratio of 5:1. The liquefaction rate was 82.8% under the above optimal conditions. Compared with raw materials, the cellulose content, hemicellulose content, and lignin content of liquefaction residue reduced by 17.90%, 9.19%, and 15.99%, respectively.

Keywords: Eucalyptus; Bark; Chemical Composition; Liquefaction

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1. Introduction

Eucalyptus globulus, a plant of *Eucalyptus* in Myrta-ceae, is a tall evergreen tree with gray-blue bark, flake peeling, and slightly angular twigs. The young leaves are opposite, the leaves are oval, the base is heart-shaped, sessile, and powdery. They mainly grow on the plateau in Southwest China (Sichuan, Chongqing, Guizhou, Guangxi, Yunnan, and Tibet). *Eucalyptus globulus* grows facing the sun, has strong adaptability, fast growth, poor damp heat resistance, and anti-pollution. It is mostly used as street trees and afforestation trees. It is suitable for living at low altitudes and high-temperature areas. It can withstand sub-zero temperatures. The wood is widely used, but it is slightly distorted and has strong corrosion resistance. It is especially suitable for shipbuilding and wharf materials; Flowers are nectar plants; the oil content of leaves is 0.92%, which can be used to make white tree oil for medicinal purposes. It can strengthen the stomach, stop neuralgia, treat rheumatism and sprain; It is also used as insecticide and disinfectant. It has a bactericidal effect^[1].

The data show that by the middle of the 21st century, the world will have a fossil energy crisis. It is of great significance to use modern high-tech biomass energy development and utilization technology

to convert environment-friendly biomass materials into clean, economical, and renewable biomass energy^[2]. *Eucalyptus* is widely distributed in China, and the plantation area has reached 1.7 million hm². As an important part of biomass materials, *Eucalyptus* grows very fast. It is a good renewable resource. As an excellent energy tree species, *Eucalyptus* is known as a “petroleum plant”. *Eucalyptus* is widely used, but its comprehensive level of processing is low. In the production, a large number of *Eucalyptus* processing residues are directly thrown away or burned, which not only causes environmental pollution but also wastes a lot of biomass resources. This paper makes a basic exploration of the liquefaction process of *Eucalyptus globulus* bark. Transforming the waste *Eucalyptus globulus* bark into reusable biological small molecular substances through the liquefaction process is not only conducive to alleviating the energy crisis, protecting the environment and reducing waste but also laying a foundation for the industrial application of biomass materials. Liquefaction is an effective way to make full use of the potential value of natural wood materials. Liquefied products can be used in fuel oil, adhesives^[3–6], foam and molding materials and other fields, and have wide application prospects^[7–10]. In this study, glycerol was used as a liquefaction agent, and concentrated sulfuric acid and concentrated phosphoric acid as catalyst. The effects of temperature and catalyst dosage on the liquefaction behavior of *Eucalyptus globulus* bark were discussed. The optimal liquefaction process of *Eucalyptus globulus* bark and its effects on the main chemical components of liquefaction products were obtained.

Scholars at home and abroad have also done some research on liquefaction. Some scholars use phenol as a liquefaction agent, such as Bai shixinfu, Lin Zhenlan, Alam, Doh, etc. Others use polyhydroxy alcohol as a liquefaction agent, such as Kurimoto, Li Wenzhao, Yamada, etc.^[11]. They all use peg as liquefaction agents and liquefaction process parameters. In this study, glycerol is selected as a liquefaction agent, because it is considered as a by-product of alcohol production from crop straw for its low price, economical efficiency, and applicability.

ty.

2. Experiment materials

Take 5 5-year-old *Eucalyptus globulus* with uniform DBH from the campus of Sichuan Agricultural University, take some bark, pulverize, pass the standard sample sieve, and retain 40–60 mesh samples. Place the prepared samples in an electric oven (103 ± 2 °C) for drying for 48 hours. After drying, put them into closed self-sealing bags for standby.

3. Experiment method

3.1 Extraction of phenol

Prepare benzene alcohol mixture (toluene: 95% ethanol = 1:2 for standby) by using the method described in GB 2677.6-1994. Weigh 2.00 g–2.005 g (m_0), and wrap the absolutely dry test material with quantitative filter paper. Then put them into the Soxhlet extractor, and add 225 mL of benzene alcohol mixture. Connect 250 mL round bottom flask under Soxhlet extractor, and time from boiling. Extract for 4 hours until the benzene alcohol in extractor becomes colorless, take it out, recover benzene alcohol from the mixture in flask by rotary evaporation, and then put the flask into the oven at $103 \text{ °C} \pm 2 \text{ °C}$ for drying and weighing (m_1), flask mass (m_2). Collect the extracted test material for standby.

Content of benzene alcohol extract:

$$W_1 = \frac{(m_1 - m_2)}{m_0} \times 100\% \quad (1)$$

3.1.1 Determination of nitric acid-ethanol cellulose content

1) Nitric acid ethanol configuration: use concentrated nitric acid and anhydrous ethanol. According to the volume ratio of 1:4, first add 400 mL of ethanol into 1,000 mL beaker, and then add 100 mL of concentrated nitric acid in 10 times. Stir continuously during each addition.

2) The content of cellulose was determined by nitric acid ethanol method. G4 glass sand core funnel was burned at 500 °C to a constant mass; Accurately take 1.000–1.005 g (m_0) of absolute dry sample; put it into a 250 mL clean and dry round bottom flask.

Add 25 mL nitric acid ethanol mixed solution, put it into a constant temperature water bath pot. Then put a reflux condenser on it, and heat it in a boiling water bath for 1 h. Take out the solution, use G4 sand core funnel to filter the solvent through vacuum water circulation, repeat three to five times until the fiber dimension turns white, and wash it with 10 mL nitric acid ethanol mixed solution, Wash it with hot water until it is not acidic. Finally, wash it twice with absolute ethanol, drain the filtrate, put the G4 sand core funnel containing residue into the oven, dry it at 103 ± 2 °C until the mass is constant, weigh the mass (m_1), then place the sand core funnel in the horse boiling furnace, burn it at 500 °C until the mass is constant and weigh (m_2).

Cellulose content:

$$W_2 = \frac{m_1 - m_2}{m_0} \times 100\% \quad (2)$$

3.1.2 Determination of hemicellulose content

According to the method described in GB 2677.5-1991, solid caustic soda is used as the chemical reagent, which is prepared by a mass fraction.

Experimental steps: weigh 1.000 g–1.005 g (m_0) of the absolute dry material after benzene alcohol extraction, put it into a 250 mL round bottom flask, add 150 mL 2% NaOH solution, put it into a constant temperature water bath, heat it at 80 °C for 3.5 hours, take it out, filter it with a Buchner funnel, wash it until it is colorless, then rinse it with deionized water, take out the residue to the Petri dish and put it in the oven, Dry at 103 ± 2 °C and the mass is m_3 . The mass of filter paper is m_1 , the mass of Petri dish is m_2 .

Hemicellulose:

$$W_3 = \frac{(m_0 - m_3 + m_1 + m_2)(1 - W_1)}{m_0} \quad (3)$$

3.1.3 Determination of lignin content

According to the method described in GB 2677.8-1993, concentrated sulfuric acid and distilled water are used as reagents and configured by volume ratio.

Experimental steps: take 1.000 g–1.005 g (m_0)

benzene alcohol extracted absolute dry material, and put it into a 50 mL conical flask. Add 15 mL of 72% concentrated sulfuric acid with a magnetic rotor, put the conical flask on a magnetic stirrer, stir for 2 hours. Wash the liquid out of a 1,000 mL round bottom flask with 560 mL distilled water, boil it on the heating jacket, and place a condensation reflux pipe on it. After 4 h, vacuum filter with Buchner funnel, wash with 500 mL hot water and then transfer the residue into the Petri dish. Put it into the blast drying oven at 103 ± 2 °C and dry it to absolute dry weight m_1 , the mass of the Petri dish is m_2 and the mass of the filter paper is m_3 .

Lignin content:

$$W_4 = \frac{(m_1 - m_2 - m_3)(1 - W_1)}{m_0} \times 100\% \quad (4)$$

3.1.4 Extract of 1% NaOH

According to the method described in GB 2677.5-1991, prepare NaOH with a mass fraction of 1%. Weigh 2.000 g–2.010 g (m_0) of absolute dry material, and put it into a 250 mL flat bottom flask. Add 100 mL of sodium hydroxide solution, and boil it in a constant temperature water bath for 1 h. Take out vacuum suction filtration, then add 100 mL of distilled water and dry it. Add 50 mL of 10% glacial acetic acid, filter it again with distilled water until it is colorless and tasteless. And then transfer it to the Petri dish, put it into the blast drying oven, dry it at 103 ± 2 °C for 12 h, and weigh it to obtain (m_1), the weight of filter paper (m_2), and the Petri dish (m_3).

Extract content:

$$W_5 = \frac{m_0 - (m_1 - m_2 - m_3)}{m_0} \times 100\% \quad (5)$$

3.1.5 Hot water extract

According to the method described in GB 2677.4-1993, take 2.000–2.010 g (m_0) of absolute dry material and put it into the Soxhlet extractor. Add 225 mL of distilled water, put a circulating condensing device on it, put down a 250 mL round bottom flask, and boil it on the heating furnace for 4 hours until it loses color. Put the residue into the Petri dish to dry and weigh (m_1), the mass of Petri dish

(m_2) and the mass of filter paper (m_3).

Hot water extract content:

$$W_6 = \frac{m_0 - (m_1 - m_2 - m_3)}{m_0} \times 100\% \quad (6)$$

3.1.6 Determination of ash content

According to the method described in GB 2677.3-1993, clean the crucible, put it into the horse boiling furnace to burn until the mass is constant, cool, weigh and count (m_1). take 2.000 g–2.010 g (m_0) of absolute dry material, put it into the crucible, put it into the horse boiling furnace, keep it at 600 °C for 0.5 h, burn until the mass is constant, and weigh (m_2).

Ash content:

$$W_7 = \frac{m_2 - m_1}{m_0} \times 100\% \quad (7)$$

3.1.7 Liquefaction method

Chemical agent: concentrated sulfuric acid (mass concentration: 98%; high-quality pure GR; Sichuan Xilong Chemical Co., Ltd.); Methanol (analytical pure AR; Industrial Development Zone, Mulan Town, Xindu District, Chengdu); Glycerol (analytical pure AR; Chengdu Kelong Chemical Reagent Factory); Dimethyl silicone oil (analytical pure AR; Industrial Development Zone, Mulan Town, Xindu District, Chengdu).

Instrument and equipment: df-101s collector type constant temperature heating magnetic stirrer (Gongyi Yuhua Instrument Co., Ltd.); 101a-3 electric blast drying oven (Shanghai Experimental Instrument Factory Co., Ltd.); Electronic balance (E = 10 d; Beijing sedolis Instrument System Co., Ltd.); 150 mm Brinell funnel, SHZ—III circulating water vacuum pump (Zhejiang yellow Yan Liming Industrial Co., Ltd.).

Experimental steps: weigh 50 g of glycerol into a round bottom flask, add 1.5 g of concentrated sulfuric acid/concentrated phosphoric acid as catalyst, put it into the flask when the oil bath temperature reaches the ideal temperature and put a rotor in the flask. When the glycerol reaches the required temperature, take 10.000 g–10.010 g (m_0) of dry mate-

rial into the flask, and start timing after the material is completely wet. When the time is up, take out the flask, transfer the liquefied substance into the beaker with methanol, and put it on the magnetic stirrer for stirring. After mixing, filter with a Brinell funnel, wash with methanol until the solution is colorless, transfer the residue to a Petri dish, mass (m_1), the mass of filter paper (m_2), and put it into an oven to dry at 103 ± 2 °C to obtain m_3 . The filtrate from the suction bottle recovers methanol and separates polyol liquefaction products through a rotary evaporator.

Liquefaction residue rate:

$$W_8 = \frac{m_3 - m_1 - m_2}{m_0} \times 100\% \quad (8)$$

4. Design of experiment

In order to analyze the effects of liquefaction time, catalyst, and on the chemical composition of *Eucalyptus globulus* bark, the temperatures of liquefaction experiment are set as 80 °C, 100 °C, 120 °C, 140 °C, 160 °C and 180 °C respectively, and the catalysts are concentrated sulfuric acid and concentrated phosphoric acid respectively. The effects of different catalysts on the liquefaction rate of *Eucalyptus globulus* bark and the chemical composition of liquefaction residue under the same temperature are compared and analyzed, To explore the best liquefaction process conditions.

5. Results and analysis

5.1 Chemical composition analysis of *Eucalyptus* bark

The main components of *Eucalyptus globulus* bark were determined, such as cellulose, hemicellulose and lignin. The experimental results show in **Table 1**. Comparing the contents of cellulose, hemicellulose, and lignin in *Eucalyptus globulus* bark, the content of lignin in *Eucalyptus globulus* bark is the highest, accounting for 45.37%, followed by cellulose and hemicellulose, with the contents of 35.65% and 18.98% respectively. *Eucalyptus globulus*'s contents of 1% NaOH extract and ash in the bark were 23.64% and 7.50%, respectively. The content of ben-

Table 1. Analysis of chemical components of *Eucalyptus globulus*'s bark

Component	Benzene alcohol extract	Cellulose	Hemicellulose	Lignin	1% NaOH extract	Ash	Hot water extract
Content/%	10.30	35.65	18.98	45.37	23.64	7.50	7.15

zene alcohol extract in *Eucalyptus globulus* bark was higher than that of hot water extract.

5.2 Effect of temperature on liquefaction rate of *Eucalyptus globulus* bark

As can be seen from **Figure 1**, with concentrated sulfuric acid as catalyst, the liquefaction rate of *Eucalyptus globulus* bark increases with the increase of liquefaction temperature; When the temperature was 80 °C, the residue rate of *Eucalyptus globulus* bark was 70.6%. When the temperature rose to 180 °C, the residue rate was 17.8% and the liquefaction rate was 82.2%. When the liquefaction temperature is 160 °C, the residue rate of *Eucalyptus globulus* bark is 18.52%. Compared with the residue rate at 160 °C, the residue rate at 180 °C decreases by only 0.72%, indicating that the liquefaction rate of *Eucalyptus globulus* bark at 160 °C is relatively stable. Considering the economy of the test, the best liquefaction temperature is 160 °C. The experimental results show that the best liquefaction process parameters are: liquefaction temperature 160 °C, 3% concentrated sulfuric acid, solid-liquid ratio 1:5, time 60 min, and liquefaction rate can reach 81.48% (**Figure 1**). Under the catalysis of concentrated phosphoric acid, the higher the temperature, the lower the residue rate is, and the higher the liquefaction rate is. The highest occurs at 180 °C, the liquefaction time is 60 minutes, the solid-liquid ratio is 1:5, and the liquefaction rate can reach 50.736%.

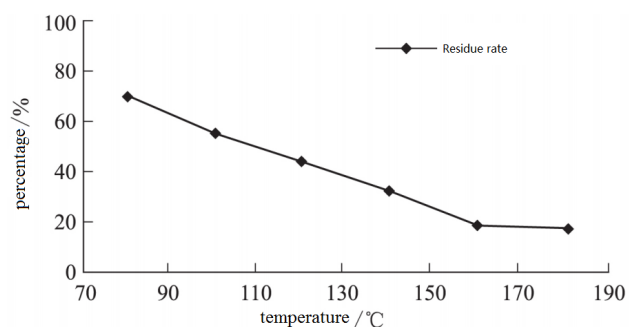


Figure 1. Liquefaction residue rate at different liquefaction temperatures with concentrated sulfuric acid as catalyst.

It shows in **Figure 1** that when concentrated

sulfuric acid is as the catalyst and the liquefaction temperature is 80 °C, the liquefaction residue rate of *Eucalyptus globulus* bark is 70.60%. After 80 °C, the residue rate decreases by more than 10% every time the temperature increases by 20 °C. Through regression equation analysis (regression equation $y = -0.6366x + 120.53$, $R^2 = 0.9976$), the liquefaction temperature shows a functional characteristic in the range of 80 °C to 160 °C. There is not a big difference in the liquefaction rate between 160 °C and 180 °C, which may be due to the degradation and liquefaction of lignin and hemicellulose. Due to its spatial structure, cellulose is difficult to contact with chemical reagents and undergo degradation reactions. When hemicellulose and lignin degrade to a certain extent, and their temperature does not reach the degradation conditions of cellulose, the liquefaction rate presents a relatively stable state.

It shows in **Figure 2** that under the catalysis of concentrated phosphoric acid, the higher the temperature, the lower the residue rate, and the higher the liquefaction rate. The optimum process conditions are as follows: the liquefaction temperature is 180 °C, the liquefaction time is 60 min, the solid-liquid ratio is 1:5. Then the liquefaction rate can reach 50.736%. When concentrated phosphoric acid was a catalyst, the liquefaction residue rate of *Eucalyptus globulus* bark was 93.8% at 80 °C, and when the liquefaction temperature increased to 100 °C, the residue rate decreased by less than 2%. It indicated that in the low-temperature range, the increase of temperature has little effect on the liquefaction rate. Until the liquefaction temperature increased to 140 °C, the liquefaction residue rate began to decrease significantly. When the liquefaction temperature was 180 °C, the liquefaction rate just reached about 50%.

The liquefaction residue rates of the two catalysts at different temperatures are compared. It can be seen from **Figure 3** that the catalytic effect of concentrated sulfuric acid is significantly better than that of concentrated phosphoric acid. At the starting

temperature of 80 °C, the liquefaction effect of concentrated sulfuric acid as a catalyst is significantly higher than that of concentrated phosphoric acid, and the residue rates are 70.6% and 93.8% respectively. The liquefaction residue rate of *Eucalyptus globulus* bark with concentrated sulfuric acid as a catalyst is 23.20% lower than that with concentrated phosphoric acid as a catalyst. At all temperatures of the experiment, the liquefaction rate of concentrated sulfuric acid as a catalyst is better than that of concentrated phosphoric acid under the same conditions.

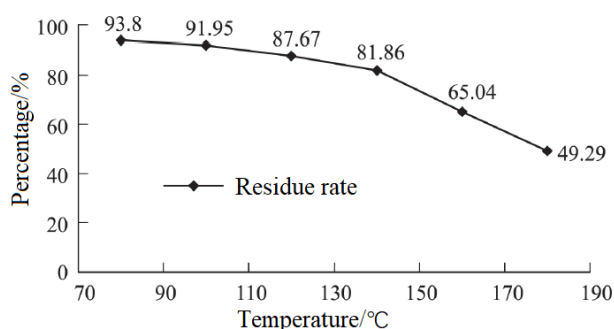


Figure 2. Liquefaction residue rate at different liquefaction temperatures with concentrated phosphoric acid as catalyst.

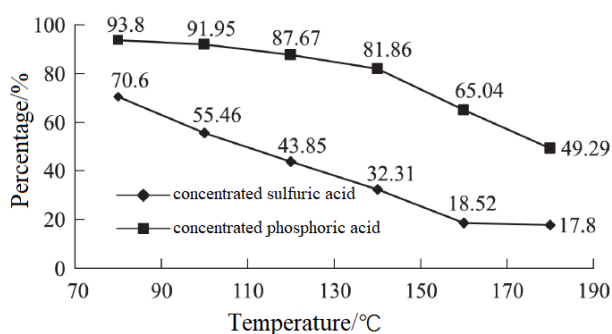


Figure 3. Comparison of liquefaction residue rates on *Eucalyptus globulus* bark with two kinds of catalysts at different liquefaction temperatures.

At 160 °C, the residue rate of concentrated sulfuric acid as a catalyst is 46.52% lower than that of concentrated phosphoric acid. The reason may be that under the catalysis of concentrated sulfuric acid, the liquefaction products polymerize and start to change from small molecules to macromolecular compounds. It can be guessed that, if the temperature continues to rise, it is possible that the residue rate will begin to increase gradually.

5.2 Effects of liquefaction on chemical constituents of *Eucalyptus globulus* bark

5.3.1 Comparison of cellulose content in *Eucalyptus globulus* bark after liquefaction with different catalysts

It shows in **Figure 4** that after liquefaction at different temperatures, the content of cellulose in *Eucalyptus globulus* bark decreases under the catalysis of concentrated sulfuric acid and concentrated phosphoric acid. When the liquefaction temperature is 80 °C, the cellulose content of *Eucalyptus globulus* bark after liquefaction with concentrated phosphoric acid as a catalyst is 33.42%. After liquefaction with concentrated sulfuric acid as a catalyst, the cellulose content of *Eucalyptus globulus* bark after liquefaction is 25.69%. Compared with liquefaction with concentrated phosphoric acid as a catalyst, the cellulose content of *Eucalyptus globulus* bark after liquefaction with concentrated sulfuric acid is lower. When the liquefaction temperature rises from 80 °C to 180 °C, the cellulose content of the two catalysts after liquefaction shows a gradual downward trend and tends to be flat at 180 °C. It may indicate that the cellulose in the bark of *Eucalyptus globulus* still gradually degrades in this temperature range. When the liquefaction temperature reaches 180 °C, the decline rate of the cellulose content of the liquefaction residue decreases significantly. This indicates that the non-crystalline region of cellulose in the possible liquefaction products has gradually degraded, and the rest is the closely arranged and regular crystalline region.

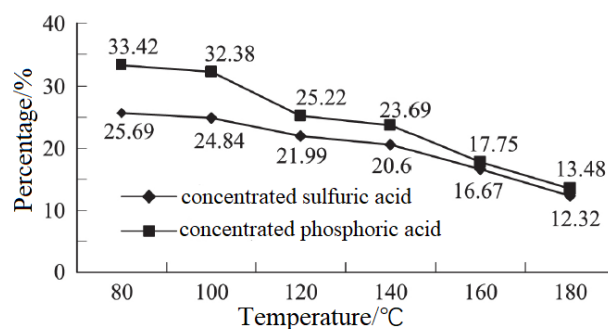


Figure 4. Effect of two kinds of catalysts on cellulose content of *Eucalyptus globulus* bark at different liquefaction temperatures.

5.3.2 Comparison of hemicellulose content of *Eucalyptus globulus* bark after liquefaction with different catalysts

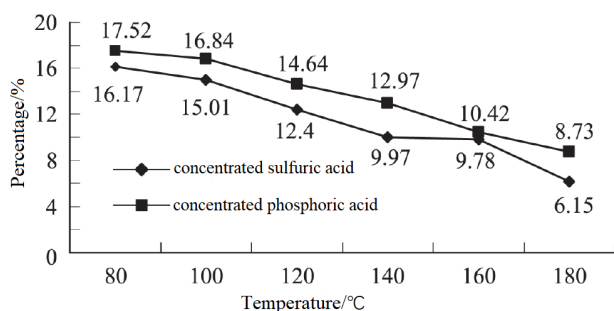


Figure 5. Effect of two kinds of catalysts on hemicellulose content of *Eucalyptus globulus* bark at different liquefaction temperatures.

5.3.3 Comparison of lignin content of *Eucalyptus globulus* bark after liquefaction with different catalysts

It can be seen from **Figure 5** and **Figure 6** that the liquefaction temperature increased from 80 °C to 180 °C, and the lignin content of *Eucalyptus globulus* bark after liquefaction with concentrated phosphoric acid and concentrated sulfuric acid as catalyst changed slightly. After liquefaction with concentrated phosphoric acid as catalyst, the lignin content of *Eucalyptus globulus* bark decreased from 34.29% to 32.58%, with a total decrease of 1.71%. Concentrated sulfuric acid as catalyst, the lignin content of *Eucalyptus globulus* bark after chemical agent liquefaction decreased from 31.41% to 27.84%, and the total decrease was 3.57%. The results showed that the catalytic effect of concentrated sulfuric acid on *Eucalyptus globulus* bark was better than that of concentrated phosphoric acid. The reason for the small total decrease of lignin content is the close benzene ring structure of lignin itself. Lignin is a three-dimensional network complex polymer composed of guaiacyl, syringyl and p-hydroxyproline through ether bond and C-C bond. In the process of liquefaction, its structure is difficult to open and react with chemical reagents.

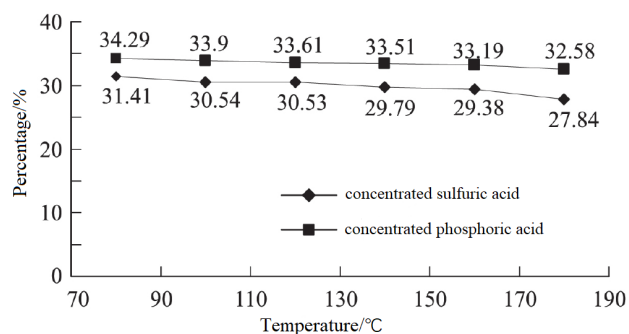


Figure 6. Effect of two kinds of catalysts on lignin content of *Eucalyptus globulus* bark at different liquefaction temperatures.

5.3.4 Comparison of main chemical components of *Eucalyptus globulus* bark before and after liquefaction

When the liquefaction temperature was 160 °C, the chemical composition of *Eucalyptus globulus* bark after liquefaction changed greatly compared with that before liquefaction. The cellulose content, lignin content, and hemicellulose content of *Eucalyptus globulus* bark residue decreased by 17.90%, 15.99%, and 9.19%. The results showed that the cellulose content of *Eucalyptus globulus* bark was most obviously affected by liquefaction, but the lignin content was the least affected by liquefaction.

Table 2. Comparison of chemical components before and after liquefaction of bark at 160 °C

Component	Before liquefaction/%	After liquefaction/%	Difference/%
Cellulose	36.65	17.75	17.90
Lignin	45.37	29.38	15.99
Hemicellulose	18.98	9.78	9.19

6. Conclusions

The cellulose content of *Eucalyptus globulus* bark was 36.65%, hemicellulose content was 18.98%, lignin content was 45.37%, benzene alcohol extract content was 10.30%, hot water extract accounted for 7.15%, 1% NaOH extract accounted for 23.64%, and ash content was 7.49%; the catalytic effect of concentrated sulfuric acid is better than that of concentrated phosphoric acid; the optimal liquefaction process obtained from the single factor experimental results of this study is as follows: with 3% concentrated sulfuric acid as catalyst, the liquefaction temperature is 160 °C, the solid-liquid ratio is 1:5, and the liquefaction rate can reach 82.8%; under

this condition, the cellulose content, lignin content and hemicellulose content of *Eucalyptus globulus* bark decreased by 17.90%, 15.99% and 9.19%

Conflict of interest

The authors declare that they have no conflict of interest.

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