

Effects of Pyrolysis Conditions on Yield of Bio-Chars from Pine Chips

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Abstract

The influences of temperature, heating rate, purge gas type, and flow rate on the yield of chars produced from pyrolyzing southern pine chips were investigated. Pyrolysis temperatures were between 450°C and 1,000°C, with heating rates of 0.5°C/min, 1.0°C/min, 10°C/min, 30°C/min, 50°C/min, and 100°C/min. Purge gases, nitrogen (N₂), hydrogen (H₂), and N₂-H₂ mixture (10% H₂), were used at flow rates from 100 to 1,000 mL/min. Pine char yield decreased as temperature, heating rate, or purge gas flow rate increased. Two regions with significantly different decrease rates of pine char yield can be identified for temperature or heating rate as they increase. The yield decrease rate turning points were 550°C and 10°C/min for yield-temperature and yield-heating rate charts, respectively. The pine char yield was lowest when hydrogen was the purge gas and highest with nitrogen.

The synthesis of novel carbon-based materials from biomass is motivated by possible applications in many fields, such as water purification, energy storage, fuel cell catalysis, bio-imaging, and drug delivery (Hu et al. 2008). The abundant, renewable, and low-cost carbon sources from biomass such as wood are an easily obtained raw carbon precursor material for value-added carbonaceous materials production. Carbon content of wood varies from about 47 to 53 percent due to varying lignin and extractives content (Ragland and Aerts 1991); almost 30 to 50 percent of the carbon in the wood is converted to solid char during the pyrolysis process. Chemical components of loblolly pine (*Pinus taeda*) wood are 42 to 46 percent glucose in cellulose; 1 to 5 percent glucose, 10 to 11 percent mannose, 7 percent xylose, 1 to 2 percent arabinose, and 1.5 to 2.5 percent galactose in the hemicelluloses; and 27 to 30 percent lignin, to give a total carbohydrate content of 66 to 69 percent by weight (Koch 1972).

Pyrolysis converts organics such as biomass into three phases: solid (char), liquid (tars, condensable vapors, etc.), and gas by heating in the absence of oxygen (Wampler 2006). Pyrolysis processes may be conventional or fast pyrolysis, depending on the operating conditions. Conventional slow pyrolysis has been applied for thousands of years in processes such as charcoal production (Demirbas 2009). In slow pyrolysis, biomass is heated to 500°C with a vapor residence time from 5 to 30 minutes. For fast pyrolysis, biomass is rapidly heated for less than 1 minute in the absence of oxygen. Fast pyrolysis processes produce 60 to 75 percent by weight liquid bio-oil, 15 to 25 percent by weight solid char, and 10 to 20 percent by weight noncondensable gases (Mohan et al. 2006).

The operating conditions, such as pyrolysis temperatures or heating rates, are key factors affecting the three fractions (Baumlin et al. 2006). For example, higher char yields are obtained at low temperatures or low heating rates than at higher temperatures or fast heating rates. Yields of the liquid phase and/or gas phase are enhanced (Bridgwater 1994). To use biomass such as wood as an alternative carbon source for producing novel carbon-based materials, studies on the effects of pyrolysis process conditions will be necessary to maximize the yields of the most economically valuable products.

In the conversion of coal to char, it has been discovered that both raw material and operating conditions such as the heating rate, the pyrolysis temperature, and the pyrolysis time influence char yield (Guerrero et al. 2005). Studies on the influence of the coal feedstock and the experimental conditions on the characteristics of the char from copyrolysis of coal and petroleum residue indicated that char yields increased as temperature and pressure increased

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(Suelves et al. 2002). The yield of chars from the pyrolysis of maize stalk decreased from 22 to 16.3 percent as the temperature increased from 600°C to 900°C. The decrease in H (hydrogen)/C (carbon) was more than twofold that in O (oxygen)/C (Fu et al. 2009). The elemental composition of char from the pyrolysis of the pinewood chips varies with the pyrolysis temperature (Demirbas 2004). Previous studies on biomass and coal pyrolysis have shown that the increase in temperature leads to decrease in char yields because the gasification reactions take place at higher temperatures (Encinar et al. 2000).

Higher temperature, smaller particle size, and increased heating rate resulted in decreased char yield from pyrolysis of agricultural residues. The cracking of the hydrocarbons with an increase in the hydrogen content was favored by a higher temperature and by using smaller particles (Zanzi et al. 2002). Wood gives more volatiles and less char than straw and olive waste. The higher ash content in agricultural residues favors the charring reactions. The higher lignin content in olive waste results in a higher char yield in comparison with straw.

The purge gas (or carrier or sweep gas) removes volatiles from the pyrolysis environment during the pyrolysis process. Usually inert gases, like nitrogen, etc., are used as the sweep gas. Sometimes the pyrolysis process is also carried out with CO₂, steam, or hydrogen as the purge gas (Khan et al. 1988). In a previous study (Kaiho and Toda 1979) it was shown that the gases used affect both the char yield and properties. The presence of H₂ during pyrolysis significantly increases the tar yield.

Purge gas flow rate is one of the most important factors that affects the char yield. Usually, biomass particles used are larger than 1 mm. As a result, there may be significant mass transfer resistance for the organic volatiles from the feedstock. Some of these volatiles may be readsorbed and recombined on the surface of the char matrix, resulting in significant changes in the char structure (Asadullah et al. 2009).

Limited documented studies are available that systematically analyzed pyrolysis process conditions such as final temperature, heating rate, and purge gas type and flow rate on the yield of bio-chars. The objective of this study was to investigate the effects of these thermal treatment conditions on the bio-char yield.

Materials and Methods

Materials and heating device

Southern pine wood chips (> 1 mm in length) were dried in an on-site dryer to a maximum moisture content of 10 percent. The moisture content of the wood was calculated using the oven-dry method; wood was dried in a ventilated oven at 105°C for 48 hours (per ASTM Standard D2395; ASTM International 1997). The oven-dry sample was then weighed. The moisture content was then calculated by the following equation:

Moisture content(%)

$$= \frac{[(\text{weight of moist wood} - \text{weight of oven-dry wood}) + (\text{weight of oven-dry wood})]}{\text{weight of oven-dry wood}} \times 100\%$$

Pyrolysis of the pine chips was carried out in a fixed-bed tubular quartz reactor with an outer diameter of 25.4 mm and a length of 120 cm. A K-type thermocouple was placed

inside the tube and embedded in the sample to measure the temperature. For each experimental run, 15 g of pine chips was packed in the middle of the quartz tubular reactor.

Experimental design

Pyrolysis processes were carried out at five temperatures (450°C, 500°C, 550°C, 650°C, and 1,000°C) with a nitrogen flow under atmosphere pressure to study temperature effects on pine char yield. For each temperature run, the gas flow rate, heating rate, and pyrolysis time were 500 mL/min, 10°C/min, and 30 minutes, respectively.

To study heating rate effects on the pine char yield, seven heating rates (0.5°C/min, 1°C/min, 5°C/min, 10°C/min, 30°C/min, 50°C/min, and 100°C/min) were performed under a nitrogen atmosphere. For each of seven runs, the gas flow rate, final temperature, and pyrolysis time were 500 mL/min, 650°C, and 30 minutes, respectively.

Three nitrogen gas flow rates (100, 500, and 1,000 mL/min) were used to study the gas flow rate effect on char yield. For each of three flow runs, the temperature, heating rate, and pyrolysis time were 500°C, 30°C/min, and 30 minutes, respectively.

Three gases (H₂, N₂, and 10% H₂-90% N₂) were used to study the purge gas effect on char yield. For each of three runs, the temperature, heating rate, gas flow rate, and pyrolysis time were 1,000°C, 50°C/min, 500 mL/min, and 30 minutes, respectively.

The yield of pine chars was calculated by

$$X\% = \left(\frac{A}{B} \right) \times 100\%$$

where A is the weight of the char after pyrolysis, and B is the weight of pine chips before pyrolysis (Maiti et al. 2006).

Results and Discussion

Temperature effect

Three phases are produced during the thermal transformation of biomass under an inert atmosphere: gas/aerosol, liquid (tars, condensable vapors, etc.), and solid (char). The operating conditions, such as pyrolysis temperature and/or heating rate, are key factors that affect the distribution among the three product fractions; under higher temperatures or fast heating rates, the biomass thermal process can produce high yields of either liquid or gas (Baumlin et al. 2006). For example, higher char yields are typically generated at low temperatures or slow heating rates (Bridgwater 1994). To determine the effect of pyrolysis temperature on the char yield, final temperatures of 450°C, 500°C, 550°C, 650°C, and 1,000°C were examined under a nitrogen atmosphere with a nitrogen flow rate of 500 mL/min and a heating rate of 10°C/min for all final temperatures. Figure 1 shows the effect of temperature on the char yield. The yield of char in pyrolysis decreased exponentially from 35.5 percent at a pyrolysis temperature of 450°C to 29.3, 26.9, and 22.1 percent at pyrolysis temperatures of 500°C, 550°C, and 1,000°C, respectively. The char is the solid residue after pinewood chip pyrolysis, and its elemental composition and structure vary with the pyrolysis temperature. Previous studies on biomass and coal pyrolysis have shown that the increased temperature leads to decreased char yield, primarily due to gasification reactions occurring at higher temperatures (Encinar et al. 2000).

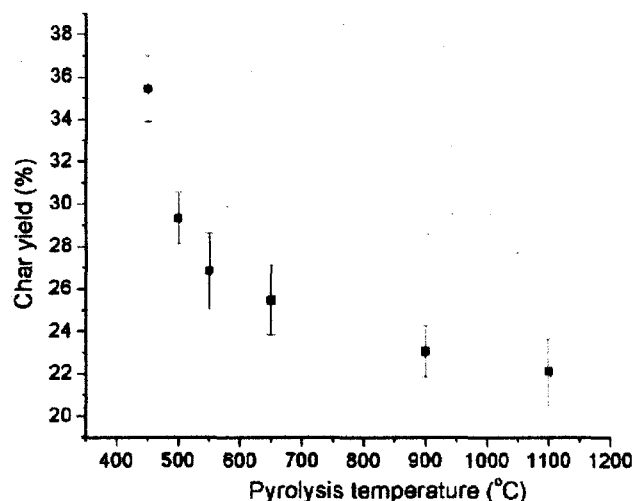


Figure 1. - Effect of final temperature on the yield of pine chars heated for 30 minutes with a nitrogen gas flow rate of 500 mL/min and a heating rate of 10°C/min.

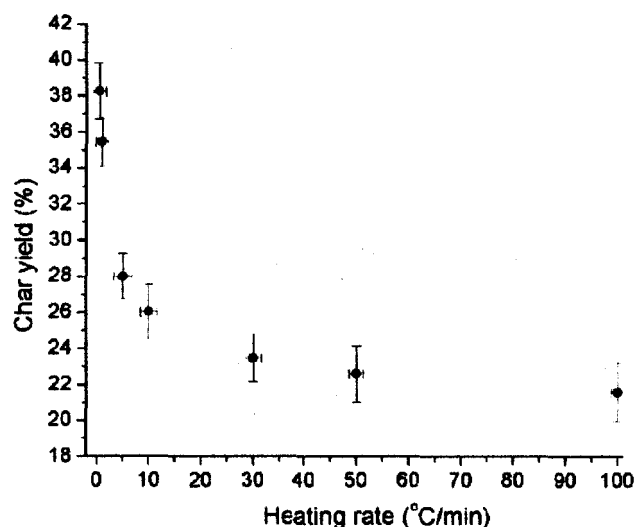


Figure 2. - Effect of heating rates on the yield of pine chars heated at a final temperature of 650°C for 30 minutes with a nitrogen purge gas flow rate of 500 mL/min.

Furthermore, the aromaticity and the carbonaceous nature of the char increased with temperature, and the char lost hydrogen and oxygen as the temperature increased (Sharma et al. 2000). The decrease in the O/C ratio was particularly large above 350°C, suggesting some decarboxylation had occurred together with dehydration (Sharma et al. 2004). The char lost both hydroxyl and aliphatic groups as the pyrolysis temperature increased, and the aromatic character increased rapidly above 450°C, resulting in an aromatic carbon content of approximately 70 percent at a high temperature. More liquid and gaseous components will be released from the pinewood chips at a higher temperature, so highly carbonized chars are produced at higher temperatures. The higher pyrolysis temperature also resulted in more liquid cracking, resulting in a higher yield of gaseous products and lower yield of tar and/or char (Zanzi et al. 2002). Under high temperatures, a portion of the volatile compounds may be decomposed to coke over the char surface or micropores in the char matrix before they are desorbed and leave the bulk of the char. Therefore, the char produced at high temperatures usually includes coking as part of the volatiles. This may be related to the fact that the rapid heating leads to a fast depolymerization of the solid material to primary volatiles, while at the lower heating rate, dehydration to more stable anhydrocellulose is limited and very slow (Chen et al. 1997).

Heating rate effect

It is known that heating rate is an important factor in determining pyrolysis product yields. The effect of heating rate on the char yield is shown in Figure 2. The heating rate in this study ranged from 0.5°C/min to 100°C/min. The nominal operating temperature is 650°C, with a pyrolysis time of 30 minutes and sweep rate of 500 mL/min nitrogen. The char yield decreased with increased heating rate; higher rates led to low char yields.

The char yield decreased abruptly from 38.3 percent at a heating rate of 0.5°C/min to 28.0 percent at a heating rate of 5°C/min, and then decreased gradually to 21.6 percent at a heating rate of 100°C/min. The effect of heating rate on weight loss during pyrolysis has been demonstrated to be

significant in a past study (Bridgwater and Peacocke 2000). Anthony and Howard (1976) investigated and compared the yields of volatile matter from a bituminous coal over a wide range of heating rates using different types of reactors and found that when the heating rate increased from 1 to 10⁴ K s⁻¹, the total yield of volatile matter increased around 11 percent. During the pyrolysis, there are several reactions/processes occurring simultaneously; among these are dehydration, decarbonization, volatilization, devolatilization, carbonization, and gasification/reforming. The strong effect of the heating rate on the formation of char from biomass may be attributed to the high cellulose content of the biomass. It is known that heating rate has a significant effect on the pyrolysis of cellulose. The dehydration of cellulose to the more stable anhydrocellulose, which gives higher char yield, is the dominant reaction at low temperatures. At low temperatures, cellulose depolymerizes, producing volatiles. If the heating rate is very high, the residence time of the biomass at low temperatures is short. Thus, a high heating rate provides a shorter time for the dehydration reactions. Less water is lost at low temperatures; hence, more cellulose and lignin will be gasified with the promotion of moisture in the wood, which leads a lower yield of char. During the pyrolysis process, water molecules will first be lost from the biomass, and then organic compounds will desorb from the biomass by volatilization processes. A portion of the volatile species may be reabsorbed and recombined onto the surface of the char matrix by the devolatilization (coking) process. At high temperatures, the gasification/reforming reaction may occur if there is some water or oxygen-containing species existing on the surface. Part of the char matrix will decompose, liberating CO, H₂, CO₂, and some light hydrocarbons. During the low heating rate pyrolysis process, there is less energy transferred to the biomass per unit time. Therefore, the structures of fewer organic compounds are broken, and fewer volatiles are desorbed from the biomass. Most of the compounds are aged and carbonized to the char matrix. Furthermore, biomass will be dehydrated at low temperatures during a slow heating rate process. Less water remains

for the gasification/reforming reaction when it reaches high temperatures. Thus, more carbon atoms are left for the char matrix. During the high heating rate pyrolysis process, more energy is provided to the biomass, and more organic compounds are active and volatilized. Also more water may be left in the biomass, leading to the gasification reaction, so the char yield is low at high heating rates. This may be related to the rapid heating, which leads to a fast depolymerization of the solid material to primary volatiles, while at the lower heating rates, dehydration to more stable anhydrocellulose is limited and very slow (Chen et al. 1997).

For the samples in the present work, the higher treatment temperatures resulted in lower yields of char and tar and higher yields of gaseous products. The temperature markedly influenced the heating rate. The energy flux is proportional to the driving force, the temperature gradient between the particle and the environment. At higher temperatures, the heat flux and the heating rate are higher. The higher heating rate resulted in decreased char yield.

Gas type effect

The purpose of the carrier gas (or purging gas or sweep gas) is to remove the volatiles from the pyrolysis environment during the biomass pyrolysis process. Usually, inert gases like nitrogen are used as the sweep gas. However, the pyrolysis process has also been carried out with CO₂, steam, or hydrogen as the purge gas. One previous effort (Kaiho and Toda 1979) has demonstrated that the type of carrier gas used affects both the char yield and properties. The presence of H₂ during pyrolysis significantly increases the yield of tar and the fluidity of coal. Therefore, use of hydrogen as the carrier gas may change a char's morphology significantly as a result of the strong association between char structure and the thermoplastic properties and the evolution of volatile matter (Khan et al. 1988). In the present study, nitrogen, hydrogen, and a mixture (10% H₂–90%N₂) were used to compare the effect of the purging gas on the char yield. Figure 3 shows the

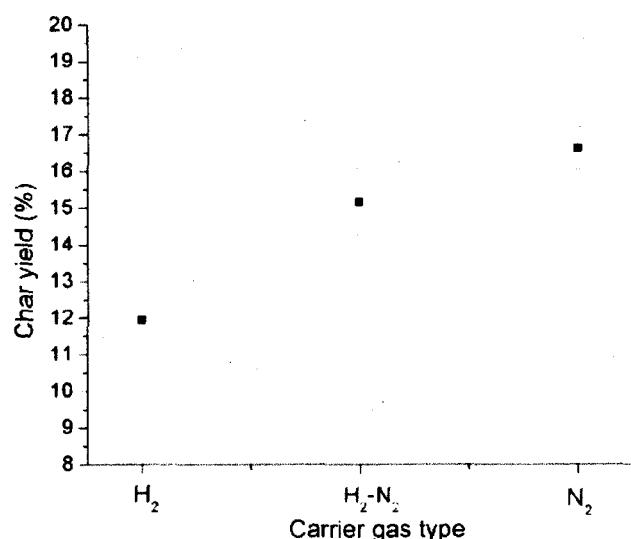


Figure 3. - Effect of purge gas type on the yield of pine chars heated at a final temperature of 1,000°C for 30 minutes with a gas flow rate of 500 mL/min and a heating rate of 50°C/min.

effect of carrier gas on char yield at 1,000°C final pyrolysis temperature with a purging rate of 500 mL/min and a heating rate of 50°C/min. The char yield was only 11.95 percent when pure hydrogen was used as the purge gas, while the yield increased to 15.17 percent when the mixture (10% H₂–90%N₂) was used. The highest yield of 16.62 percent was obtained when using pure nitrogen as the purge gas. We propose two possible explanations for why the char yield was lower with hydrogen as the sweep gas. First, hydrogen gas has the highest thermal conductivity at 0.168 W/(m·K) of all gases; it is 7 to 10 times that of nitrogen. Therefore, the heat flux in the cases where hydrogen carrier gas is used should be much higher than that where nitrogen is used; in the hydrogen case, the heating rate is relatively higher than with the nitrogen sweep gas. The higher heating rate resulted in decreased char yield. Second, reactions between the hydrogen and components in the char also led to the lower char yield when using hydrogen as the sweep gas. There are two basic reactions assumed to occur in gases containing hydrogen during the pyrolysis process:



Reaction 1 could occur in pure hydrogen or in hydrogen-containing mixtures at elevated temperatures. Reaction 2 occurs when both steam and hydrogen are present; since there is always moisture in biomass, this reaction will happen during the pyrolysis process.

Gas flow rate effect

To investigate the effect of the carrier gas flow rate on the char yield, the pyrolysis experiments for pinewood were carried out at 650°C and with different nitrogen flow rates of 100, 500, and 1,000 mL/min (Fig. 4). The nitrogen flow affected the residence time of the gas produced as a result of pyrolysis reactions by removing the products from the hot zone. This minimizes secondary reactions such as cracking and char formation (Encinar et al. 2000). Pütün et al. (2007)

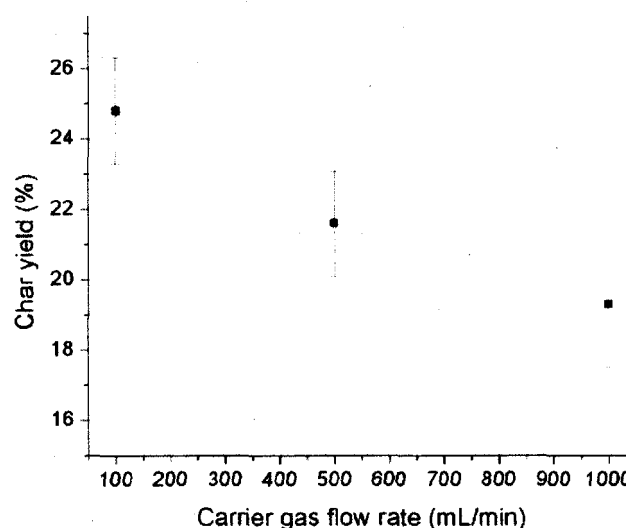


Figure 4. - Effect of purge gas flow rate on the yield of pine chars heated at a final temperature of 500°C for 30 minutes with a heating rate of 30°C/min and nitrogen gas.

observed that pyrolysis conversion increased in small amounts and there was no obvious influence on the yield of char as the rate of nitrogen increased. In the present research, the yield of the char was found to be significantly affected by the flow rate of the sweep gas. The yield of the char decreased from 24.8 percent by weight at 100 mL/min to 19.3 percent by weight at 100 mL/min. Usually, the biomass particles were larger than millimeter size. As a result, there could be significant mass transfer resistance for the organic volatiles in transfer from/onto the surface of the char particles. Some of these volatiles may be readsorbed and recombined on the surface of the char matrix, resulting in significant changes in the char structure. The deposited species may have a great influence on the char structure as well as its reactivity in the gasification step. The char structure may also influence how the species would physico-chemically deposit inside the char. As a result, the reactivity of char will change with biomass particle size. Carrier gas flow rate is one of the most important factors affecting the char yield and properties. Pyrolysis proceeds in two steps: an initial fast step followed by a slower step. It has been suggested that the primary pyrolysis reactions in the rapid pyrolysis of wood are completed within seconds, while the secondary reactions take minutes to complete. Longer residence times at high temperatures favor the secondary pyrolysis reactions, resulting in higher char yields, and also influence the composition of the gaseous products. The short residence time of the volatiles in the reactor as the nitrogen gas velocity increased caused relatively minor secondary decomposition of higher molecular weight products (Şensöz et al. 2000). Therefore, secondary reactions such as thermal cracking, repolymerization, and recondensation should be kept to a minimum for maximum liquid yield.

Conclusions

The influences of temperature, heating rate, purge gas type, and flow rate on the pine char yield were investigated. Temperature had a significant effect on the pine char yield, with char yield decreasing as the pyrolysis temperature increased. The sharp decrease in the char yield occurred at 500°C, i.e., the char yield dropped from 35.5 percent at 450°C to 29.3 percent at 500°C. The drop rate of pine char yield decreased significantly as the temperature passed 550°C toward 1,000°C.

The yield of pine chars produced under the controlled thermal treatment condition in this study was significantly affected by the heating rate. In general, the pine char yield decreased with the increase of the heating rate, and there are two different decreasing regions. The sharp decrease in the char yield occurred at the heating rate of 5°C/min compared with 0.5°C/min, i.e., the higher dropping rate of char yield occurred in the region of a heating rate from 0.5°C/min to 5°C/min. The turning point, from high to low, of the dropping rate of char yield was at the heating rate of 10°C/min. Beyond the point of 10°C/min heating rate, the char yield decreases at a significantly lower rate.

In general, the pine char yield decreased with increasing nitrogen flow rate. The purge gas type also significantly affected the pine char yield. The char yield was only 11.95 percent if hydrogen was used, while the yield increased to 15.17 percent when the mixture (10% H₂-90% N₂) gas was used, and the highest yield of 16.62 percent was obtained when using nitrogen.

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