Experimental Investigation of the Effects of Some Significant Parameters on the Pyrolysis of Solid Biomass Materials

M Alhwayzee

Department of Mechanical Engineering, College of Engineering, University of Kerbala, Iraq.
Email: Amnaalhwayzee200@gmail.com

Abstract. The effect of some important parameters on the pyrolysis process in biomass solid fuel material was investigated experimentally using a batch furnace reactor in this study. Four significant parameters were selected, and their effects on process efficiency investigated; these were process temperature, feedstock type, biomass fuel particle size, and catalyst material. The experiments were implemented using three ranges of particle size: less than 0.25 mm, 0.3 to 0.5 mm, and 0.5 to 1.18 mm; and two temperature levels: 450 °C and 550 °C. Three concentrations of catalyst material (Ceria, CeO2) by weight were investigated: 16.7%, 25%, and 35%. The composition of the produced biogases, which consisted mainly of CO and CO2, were measured using gas analyser apparatus. Each experiment was performed for a different period of time based on the produced gases; thus, the experiments each lasted between 550 and 1,000 sec. For sawdust biomass fuel, the results revealed that the composition of produced gases decreased as the range of particle size increased. Moreover, there was a considerable positive effect of the catalyst on gas composition compared to the reference case (< 0.25 mm sawdust without catalyst). An increase in catalyst percentage was also seen to affect the residence time of the biomass pyrolysis process positively. However, for wastepaper biomass, the temperature had the most considerable effect on the composition of produced gases.

Keywords: Renewable Energy, Biomass solid fuel, Pyrolysis Reactions, Particle Size, Catalyst.

1. Introduction:

Due to the gradual depletion of global reserves of fossil fuels, renewable energies are becoming more attractive. Recently, solid biomass has attracted attention as a promising source of renewable energy for bio-fuel (biochar, bio-oil, and biogas) production owing to its environmentally-friendly nature, whereby all CO2 produced by the thermochemical conversion processes can be recycled into the plant by using photosynthesis. Additionally, compared to conventional fossil fuels, biomass materials contain little sulphur and nitrogen, and thus less harmful NOx and SO2 gas emissions are produced in the biomass combustion reactions [1, 2].

Pyrolysis is one of three thermochemical decomposition processes (Combustion, Gasification, and Pyrolysis), which can convert biomass or other organic materials under heat, in various pressure (vacuum, atmospheric, and high) and inert environment (oxygen absence) conditions into solid, liquid, and gas fuel products. It thus gives a range of oils, acids, water, producer gas, and solid chars depending on the chemical composition of the biomass feedstock, reactor design, and operating conditions [3, 4]. The simplified pyrolysis reaction of complex lignocellulose molecules in solid biomass is [1, 3]
\[ C_{x}H_{y}O_{z} \text{ (biomass)} + \text{Heat} \rightarrow \text{Gases (CO + CO}_{2} + \text{H}_{2} + \text{H}_{2}\text{O} + \text{CH}_{4} + \text{C}_{2}\text{H}_{6} + \text{CxHy} \ldots \text{etc.)} \]
\[ + \text{Liquids (Tar or bio-fuel)} + \text{C (Char)} \]

As shown in the pyrolysis reaction equation, the complex and large compounds in solid biomass material decompose into simple and smaller compounds in the form of condensable vapours (tars and oil), gases, and solid char. These pyrolysis products depend on many important parameters, but of particular interest are reactor design, pressure, pyrolysis temperature, the physical and chemical properties of the biomass feedstock (such as composition, particle size, and shape), biomass heating rates, residence time, and the existence of catalyst materials [1, 2].

Temperature parameter has a considerable effect on pyrolysis performance and product type. Parbir Basu [1] showed how the biomass pyrolysis temperature affects the producer gas composition for a temperature range between 200 °C and 800 °C. At this range, H\textsubscript{2} increased considerably from 2% to 80 mol% where CO\textsubscript{2} decreased from 70% to 5%. However, the CO took on a sawtooth shape, with two peak points at 30 mol% at 200 °C, and 25 mol% at 600 °C, and hydrocarbons increased from 3 mol% at 200 °C to 47 mol% at 400 °C before decreasing gradually to 10 mol% at 800 °C [1].

The International Energy Agency (IEA) [5] reported that a temperature parameter in the range 425 °C-625 °C has a considerable effect on the yield of pyrolysis end products including biochar, bio-oil, and biogas. The results showed that at this range, biogas yield increased gradually from 6% to 37%, whereas biochar decreased from 33% to 8% while bio-oil initially rose from 55% to 75% when the temperature increased from 425 to 480 °C before reducing to 40% at 625 °C. It can be concluded that pyrolysis temperature is a significant parameter for product type specification. The results of Kantarelis and Zabaniotou's study [6] similarly showed that a temperature, between 400 and 765 °C, affected the producer gas composition of pyrolysis of cotton stalk biomass. Their results showed that CO increased from 42 to 46 vol%, CH\textsubscript{4} decreased from 13 to 8 vol%, and H\textsubscript{2} increased progressively from 18 to 38 vol%, while CO\textsubscript{2} dropped from 28 to 6 vol%. Recently, Muhammad et al [7] studied the effects of the temperature on the yields and energy recovery of pyrolysis products from co-feeding various biomass feedstocks. They concluded that the optimum yield value of biochar product was 91 wt% at 400 °C. Bio-oil and producer gas percentages were 48% at 551 °C and 22% at 600 °C, respectively. In terms of energy content, biochar decreased with increased temperature, while biogas increased; for bio-oil, the energy content trend increased up to 550 °C and then reduced.

It can thus be concluded that higher temperatures, above 450 °C, enhance tar decomposition and thermal cracking to increase the proportion of producer gas products, leading to proportionately decreased bio-oil and biochar products.

The characteristics of solid biomass (composition shape, particle size, etc.) also have effects on the products of the pyrolysis process due to their direct impact on heating rates. Smaller particles have less heat transfer resistance and less resistance to volatile gas release, thus producing a higher bio-oil yield. In contrast larger particles have higher resistance and produce a higher biochar yield [1].

Jun et al showed how particle size affects the yield and the type of pyrolysis products. They pyrolyzed different particle sizes ranging from 0.18 to 5.6 mm of oil mallee woody biomass in a fluidised bed pyrolyzer at 500 °C. They found that the liquid product yield decreased when the biomass particle size increased from 0.3 to 1.5 mm. After this size, they did not notice any additional decrease in the liquid (bio-oil) yield, however. Thus, they concluded that the small size might produce lower yields of light components of bio-oil and higher yields of heavy components than the large particles.

Siyi Luo Bo et al [9] pyrolyzed solid biomass material at 800 °C in a fixed bed reactor for three ranges of particle size (0 to 5 mm, 5 to 10 mm, and 10 to 20 mm). The results showed that the small particle size produced high yields of biogas and reduced tar and char, and that the proportions of H\textsubscript{2} and CO gases increased when particle size decreased.
Examining fast pyrolysis of biomass wood in a drop tube furnace for several seconds residence time, Santiago Septien et al [10] found no effect of particle size, which ranged from 0.35 to 0.8 mm, on the final pyrolysis products at three high temperatures (1000, 1200, and 1400 °C).

Guizani et al [11] reported that small particle size enhanced pyrolysis gas production and decreased the char yield. They further explained that the heating rate of the biomass particle, which depended on size and shape and pyrolysis operating conditions, affected the yield of pyrolysis bio-products and biochar properties.

In their experimental and numerical study, Arvind Atreya et al [12] concluded that, under high-temperature conditions, small biomass particle sizes could be pyrolyzed faster than large particles, resulting in less char and more volatile compounds.

Ceria CeO$_2$ is a crucial component of the catalyst mixture. It supplies a higher storage capacity of oxygen due to its potential to be reduced and re-oxidized. Due to this redox property, ceria can provide catalytic support for many chemical reactions [13, 14, 15]. Redox catalysts have an oxidation state feature that permits them to be used for partial oxidation of the hydrocarbons present in the producer gas from gasification and pyrolysis processes [16].

In their studies, Legonda [16], Wu, et al [17], and Jiang et al [18] found that a ceria catalyst had the potential to oxidize carbon to CO at temperatures of 300 °C and 400 °C. Further, Legonda [16] showed that in the catalyst bed, carbon was decreased when ceria concentration was increased, giving evidence that ceria offers good resistance to cocking. This finding was confirmed and reported by [17].

In the current paper, an experimental study was conducted to investigate the effects of some important parameters on the performance of pyrolysis of solid biomass materials, with a particular focus on the composition of the biogas products. Two pyrolysis temperatures were selected, 450 °C and 550 °C to be used with three ranges of pinewood sawdust biomass particle size (less than 0.25, 0.3 to 0.5 mm, and 0.5 to 1.18 mm). Finally, three concentrations of ceria catalyst material, 16.7%, 25%, and 35% by weight percent in the ceria-biomass mixture, were selected. Pyrolysis of the biomass was done using a batch furnace reactor in an inert environment using N$_2$ gas. Pyrolysis producer gas composition, particularly CO and CO$_2$, was then analysed in term of volume percent using the gas analyser.

2. Experiment Set-up

2.1 Biomass pyrolysis rig

Pyrolysis reactions of solid biomass materials were carried out using a high-temperature batch reactor rig as shown schematically in Figure 1. This rig consisted of a batch tubular reactor furnace (4 and 6). Inside this tubular reactor, a boat, holding a solid biomass reactant, was placed (5), and a combined controller was used to set and control the required reaction temperature. A cylinder of pure N$_2$ gas (1) was used to supply a specific and sufficient flow rate of N$_2$ gas measured using an N$_2$ gas rotameter. This gas was used to obtain an inert environment to enable biomass pyrolysis reactions. The producer gas was filtered at the reactor outlet using filter paper to remove all solid fine contaminants to avoid damage to downstream equipment and instruments. After that, a standard tar capturing unit (7 and 8) was used to remove all condensable vapours and tar products to avoid damage to the downstream devices. This unit consisted of four standard bottles (8) connected in series and filled with 99.8% isopropanol solvent compound. This solvent was used to screen all tar compounds. These four bottles were accommodated inside a freezer (7) with a minimum temperature of -15 °C. From the outlet of the fourth bottle, the producer gas was sent to two silica gel bottles (9) to ensure that all impurities were completely removed. Thereafter, the gas was driven to the gas analyser device (11) using a vacuum pump (10). A regulator valve (2) and flow-meter (3) were used to control the required producer gas flow rate. The gas analyser was initially purged with N$_2$ gas and then calibrated using a standard gas mixture. The composition rates of each gas, CO$_2$ and CO, were recorded as volume percent as a function of time. An extraction system was then used to exhaust the producer gas outside.
Figure 1: Schematic layout of the biomass pyrolysis rig.

1- N$_2$ gas bottle, 2- N$_2$ regulator valve, 3- Gas rotameter, 4- Furnace, 5- Boat with biomass sample, 6- Pyrolysis reactor, 7- Freezer room, 8- Impinger bottles with isopropanol solvent, 9- Silica gel bottles set, 10- Vacuum pump, 11- Gas analyser device.

2.2 Preparation of biomass material

For this study, a biomass material called pinewood sawdust was selected as a solid reactant for pyrolysis reactions. This commercial pinewood sawdust was supplied by a commercial British supplier company called Batleys, and the particle size (as received) was typically 3 mm, as shown in Figure 2.

Figure 2: Pinewood sawdust sample (3 mm as received).

Three different ranges of particle size for this biomass were then prepared: less than 0.25 mm, 0.3 to 0.5 mm, and 0.5 to 1.18 mm. These selected sizes offered a wide range of biomass particle sizes for comparing and studying effects on the pyrolysis process. A Knife Mill of type FRITISH-Germany was used to cut the supplied pinewood biomass into different sizes using special curvature mesh trays to reduce raw particle size within a required range. The ground biomass material for a specific mesh size was sieved to desired size using standard sieving trays. The sieving analysis procedure from BSI (Anon 2011b) was used [19], and sieve apertures 0.300 mm, 0.500 mm, and 1.18 mm were thus employed for biomass sieving. The properties of the test pinewood biomass were expressed as proximate, ultimate analysis, and GHV, as described in Table 1 [16].
A pure material called Ceria (CeO₂) was used as a catalyst to study its effects on the acceleration and enhancement of the pyrolysis reactions. It was mixed uniformly with the pinewood sawdust biomass in different concentrations by weight percent (16.7%, 25%, and 35%).

2.3 Biomass pyrolysis experiment procedure

After the temperature reached the required reaction temperature, a specific N₂ gas flow rate was permitted to flow inside the reactor, and a vacuum pump was switched on to draw the pyrolysis product gases through the tar capture system to the gas analyser. Then, 50 g of the specified range of particle size of biomass material was weighed out and placed on the boat holder, which in turn was placed inside the tubular reactor furnace. At this time, t=0.0, the pyrolysis reactions began, and the concentration of producer gases, especially CO and CO₂ gases, were recorded in terms of volume percent. For one specified particle size, two reaction temperatures were selected, 450 °C and 550 °C for temperature effect studying. Also, as mentioned above, three particle size ranges of biomass (less than 0.25 mm, 0.3 to 0.5 mm and 0.5 to 1.18 mm) were used to test the effect of biomass particle size on the performance of the pyrolysis process. Finally, three experiments for three concentrations of Ceria (CeO₂) catalyst material-biomass mixtures in weight percentages 16.7%, 25%, and 35%, were conducted at a specified temperature and biomass particle size. The gases composition results were recorded using a laptop video recorder.

3 Results and Discussion

3.1 Effect of Pyrolysis Temperature

Figures 3 and 4, a and b, present the experimental results of the producer gas composition, represented by CO and CO₂ gases throughout the pyrolysis reaction time, based on the pyrolysis of pinewood sawdust and waste paper biomass for two operating temperatures, 450 °C and 550 °C. Figures 4 and 6, a and b, highlight the effect of pyrolysis operating temperature on producer gas composition and pyrolysis time for both biomass materials.

In general, Figures 3 and 4, a and b, show, the trend of the producer gas distribution of CO and CO₂ within the pyrolysis period takes on a normal distribution. This trend may be due to the batch operation of pyrolysis experiments. Initially, the amount of the released gases increases sharply, and when it reaches peak point, this amount decreases as a result of the conversion of the solid biomass

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Table 1: Proximate, ultimate analyses and GHV for pinewood biomass material as received (ar) basis [16].

| Analysis                     | Weight Percent |
|------------------------------|----------------|
| **Proximate analysis**       |                |
| Moisture Content             | 02.47          |
| Volatile Matters             | 82.73          |
| Ash Content                  | 00.43          |
| Fixed Carbone                | 14.37 (by difference) |
| **Gross Heating Value (GHSV)** | 19.09 (MJ/kg)  |
| **Ultimate Analysis**        |                |
| C                            | 49.4           |
| H                            | 05.9           |
| S                            | 00.02          |
| N                            | 00.3           |
| Cl                           | N/A            |
| O                            | 40.68 (by difference) |
reactant. This peak point can be taken as an equilibrium (steady-state conditions) amount if the operation is continuous. These results agree with the findings of other studies [1 and 5] and confirm that the non-condensable permanent gases such CO and CO$_2$ are mainly produced as a result of the gas-phase reactions.

a) at 450 °C
b) at 550 °C

Figure 3: Producer gas composition vs pyrolysis time for pinewood sawdust biomass pyrolysis: a) at 450 °C and b) at 550 °C.

Furthermore, from Figures 5 and 6, a and b, it can be observed that the operating temperature parameter has a considerable effect on both producer gas composition and pyrolysis time for both solid biomass materials. For pinewood biomass, when the temperature increased from 450 °C to 550 °C, CO and CO$_2$ concentrations and pyrolysis time at each peak point were significantly increased, from 0.85 to 4.2 vol% for CO and 1.2 to 3.9 vol% for CO$_2$, respectively, and from 164 to 530 secs for CO and 179 to 551 secs for CO$_2$, respectively. For the waste newspaper, the increase was from 0.3 to 0.85 vol% for CO and from 0.4 to 0.91 vol% for CO$_2$, respectively, whereas for the pyrolysis time, the temperature effect was approximately steady at 136 secs for CO and 160 secs for CO$_2$. Another observation, seen in Figure 4-a, shows that the amount of CO$_2$ gas is greater than that of CO at 450 °C, while this is reversed at 550 °C. A possible explanation for this behaviour might be that
endothermic dry reforming reaction is enhanced at 550 °C, producing more CO gas. The partial oxidation reaction of hydrocarbon compounds might be enhanced at this temperature as well.

From these findings, for pinewood sawdust biomass, it can be concluded that the pyrolysis temperature at 550 °C has a more significant effect than that of 450 °C in terms of producer gas composition. However, pyrolysis time demonstrated the opposite outcome. The former effect may be attributed to the fact that high temperature promotes and accelerates the rate of all chemical, endothermic, and exothermic reactions, as well as providing higher heating rates, especially for pyrolysis reactions, allowing more volatile matter to be released, and the secondary reactions to be thus enhanced. These results and interpretations are consistent with those of other studies \[7, 20\], and all these reactions affect the producer gas yield positively. The lower temperature, 450 °C, had insufficient potential to enhance those reactions and took too much time to release product gases. For waste newspaper biomass, the negative finding might be attributable to the poor characteristics, based on proximate and ultimate analysis, of this biomass feedstock. However, this result has not previously been described.
3.2 Effect of biomass particle size

The effects of particle size of pinewood sawdust biomass material on the composition of CO₂ and CO gases are shown in Figure 7, in a and b, respectively. Three ranges of particle size were used: less than 0.25 mm, 0.3 to 0.5 mm, and 0.5 to 1.18 mm.

From Figures 7, a and b, it can be seen that the pyrolysis of the biomass sawdust gives the same trends for both CO and CO₂ gases. In the first period, before the peak point, CO is released slightly before CO₂. This difference increases at peak point and just after. This behaviour can be interpreted as fixed carbon in the biomass being oxidized based on its oxygen content under pyrolysis conditions before initially being converted into CO and CO₂ gases. At peak point and after, for a short time, fixed carbon remains, and some of CO are oxidized and converted to CO₂.

This behaviour can be attributed to the large size of these biomass particles, which might create an inhomogeneous settling of particles on the boat, causing high voids with air environments that would promote rapid pyrolysis reactions.

The overall effect of biomass particle size on the pyrolysis producer gas composition and pyrolysis time is summarised in Figure 8, a and b, respectively, where the peak points of the composition and their times of each gas, CO and CO₂, were chosen as seen in Figures 3, 4 and 5.
Figure 8: Effect of particle size of pinewood sawdust biomass on a) the gas composition of pyrolysis producer gas and b) pyrolysis time at 550°C.

Figure 8-a illustrates that the composition of the two gases decreases as particle size range increases, whereas Figure 8-b shows that pyrolysis time increases. For gas composition, this effect may be attributed to several things: 1) Higher surface area in small size particles compared large size, with the smaller particles having faster heating rates due to their larger surface area; 2) For smaller particle sizes, the main control step in the pyrolysis process is reaction kinetics, while for larger particle size, the gas diffusion step is controlled because the product gas generated inside the particle is not easily diffused out [21, 22]; and 3) While for pyrolysis time, the particle size effect can be interpreted as the pyrolysis process being dominated by the heat transfer process, both particle size and shape affect the conversion time significantly, especially for large size particles [23]. These conditions lead to high pyrolysis reactions producing high-quality gases. Previously, [24] attributed this effect to the higher temperature gradient for large particles compared to small particles at a given time leading to an increase in char and a decrease in gases. Another observation of Figure 8-a is that there is an overlap in the data at particle size 0.5 to 1.18. This point was discussed in detail in section 3.1, at the end of paragraph three.

3.3 The Effect of CeO2 catalyst concentration

The effects of the CeO2 catalyst amount, which was added to < 0.25 mm pinewood sawdust biomass by weight percent, on the producer gas composition of CO2 and CO are shown in Figure 9, a and b, for four weight percentages of catalyst (0.0%, 16.7%, 25%, and 35%).
Figure 9: Composition of CO2 (a) and CO (b) pyrolysis gases produced from four different mixtures, 0.0, 16.7, 25, 35 wt%, of CeO2 catalyst + < 0.25 mm pinewood sawdust biomass at 550 °C.

From this figure, it can be seen that the trend of the producer gas distribution of CO2 and CO throughout the pyrolysis period takes on similar behaviour to that explained in the temperature effect section above.

Figure 10: Effect of catalyst concentration in pinewood sawdust biomass on, a) gas composition and b) Pyrolysis time, at 550 °C.

Figure 10, a and b presents the effect of the catalyst amount in the catalyst-biomass mixture on the producer gas composition and pyrolysis time, respectively. From Figure 10-a, it can be observed that the gas compositions increase gradually from 4.8 to 6 vol% for CO and slightly from 5.1 to 5.6 vol% for CO2, when the catalyst weight percentage increases from 0.0% to 16.7%. The gas compositions decrease considerably to 5.3 and 3.4 vol% for CO and to 5.1 and 3.8 vol% for CO2, when the catalyst percentage increases to 25% and 35%, respectively. Similar behaviours can be observed for pyrolysis time, as shown in Figure 10-b. The catalyst effect on pyrolysis time is thus clear at 25% and 35%, where the time is reduced to 94 and 83 secs, while the effect on gas compositions is evident at 16.7%, with 6 vol% for CO and 5.6 vol% for CO2 at 240 and 255 secs, respectively.

Finally, it can be concluded that there is a considerable positive effect of the catalyst on the CO gas composition at low catalyst concentrations of between 0.0% and 25%. A similar trend is observed in pyrolysis time but the highest two catalyst concentrations, 25% and 35%, also positively affect this,
and the time is considerably reduced. Thus, optimal operating conditions, including specified ceria loading, are necessary.

The higher concentrations of CO gas compared with CO$_2$ gas at catalyst loading, especially at 16.7% and 25%, can be attributed to the existence of reduced cerium oxide, Ce$_2$O$_3$, which reacts rapidly with CO$_2$ to produce CO [16]. There is also an opposing oxidation reaction for CO converting to CO$_2$ due to the high oxygen concentration in the biomass feedstock, as shown in Table 1. This might affect the balance between CO and CO$_2$ concentrations. The oxidation reaction of carbon C by the CeO$_2$ catalyst at these operating conditions also provides another possible source of CO increase in the product gases. A similar conclusion was proposed based on the ability of ceria to oxidize carbon at temperatures between 300 °C and 400 °C [25, 26].

4 Conclusion

Four essential parameters, process temperature, feedstock type, biomass fuel particle size, and catalyst material, were studied to investigate their effects on biomass pyrolysis performance, especially in terms of producer biogas composition. The results showed that temperature positively and considerably affects producer gas composition when increased from 450 °C to 550 °C for pinewood sawdust biomass, whereas for waste newspaper biomass, this effect was not observed. A similar considerable impact on pyrolysis time was noted. Biomass particle size showed a noticeable impact on biogas composition for three ranges of particle size, < 0.2, 0.3 to 0.5, and 0.5 to 1.18 mm. The largest size had a greater influence on pyrolysis time than the smaller sizes due to the low heating rate of larger particle sizes. Finally, 16.7 wt% of ceria CeO$_2$ catalyst provided a positive effect on producer gas composition compared to other tested catalyst concentrations of 0.0, 25, and 35 wt%.

5 Nomenclature

BSI = British Standard International
C = Carbone atom
CH$_4$ = Methane gas
Cl = Chlorine gas
CO = Carbon monoxide gas
CO$_2$ = Carbon dioxide gas
H = Hydrogen atom
H$_2$ = Hydrogen gas
LHV = Low heating value, MJ/kg
N = Nitrogen atom
N$_2$ = Nitrogen gas
O = Oxygen atom
O$_2$ = Oxygen gas
S = Sulphur atom
wt% = Weight percentage

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