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Optimization of energy valorization from lignocellulosic biomass and their pure woody pseudo-components using a green technology

N. Boukaous[a,b], L. Abdelouahed[b], M. Chikhi[a], B. Taouk[b], A-H. Meniai[a] and B. Kebabi[c]

[a]*Faculty of Process Engineering, University of Constantine 3, Constantine, Algeria*

bSafety Laboratory of Chemical Processes, Normandy Univ., INSA Rouen Normandy, UNIROUEN, Rouen, France

cLaboratory of Pollution and Water Treatment, Faculty of Exact Sciences, Department of Chemistry, University of Constantine 1 Frères Mentouri, Constantine, Algeria

*Corresponding author, email: nourelhouda.boukaous@insa-rouen.fr

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Abstract

Optimization of air-gasification technology of the lignocellulosic biomass Cistus monspeliensis (CM) and the pure woody pseudo components (cellulose, hemicellulose and lignin) was realized by amplifying experiments in a semi-batch reactor. For each single sample, the effect of the both temperature (750-900°C) and air fraction in nitrogen (0%-48%) on the gasification performance including gas product yield and gas chemical composition were discussed in depth. The results indicated that the increase of gasification temperature and air fraction was found to increase the production of gas during the gasification process of biomass, cellulose, hemicelluloses and lignin. CO, CO_2, H_2 and CH_4 are main components of the gas phase. 4% of air was found to be the optimal fraction in the gasification of CM and this value was applied in the gasification of cellulose, hemicellulose and lignin. The gas composition with the highest CO production was obtained from cellulose (48%-51%) at 0% air, while lignin produced more hydrogen and less CH_4 than others.

Keywords: Air-gasification, Cistus monspeliensis, semi-batch reactor; air fraction

1. Introduction

The world's reserves of non-renewable energy resources (oil, coal, gas) are decreasing ineluctably. As well as the current use of these sources has generated environmental problems, and has caused pollution in air, water and soil over the years. These latter negative effects have attracted attention of developing new technologies in order to obtain clean and abandoning energies through the utilization of renewable resources [1]. The ownership of biomass, as the only organic matter-containing source compared to other renewable sources, makes it the only one able to produce three fractions liquid, solid and gaseous [2]. Biomass gasification is an effective alternative over other conversion methods, which offer high economic benefits and great potential for development and utilization [3,4].

Gasification is the thermochemical technology that converts carbon-containing materials, including coal, waste and biomass into hydrogen and other valuable gases which in turn can be used to produce electricity, liquid bio-fuels such as methanol and Fischer-Tropsch synthesis motor fuels and other valuable products [5–8].The conversion process occurs at high temperatures under air/O_2, or water vapor or carbon dioxide, or combination of these gases as the gasification agent.

Generally, the mechanisms of biomass gasification characterizes by its complexity because of its grouping of different reaction in the gasifier. It includes humidity evaporation pyrolysis, combustion, gasification of volatiles and char gasification [9]. Moreover the rate gasification of char is dependent on gasifying agents, so char rate is considered as the rate controlling step overall conversion process [10,11].

Major reactions occur during gasification are given by equations (1)-(5):

Char oxidation:
$C + O_2 \rightarrow CO_2$ +408.8kJ/mol (1)
Boudouard reactions:
$CO_2 + C \rightarrow 2\,CO$ -162kJ/mol (2)
Water-gas shift reaction:
$CO + H_2O \rightarrow CO_2 + H_2$ +41kJ/mol (3)
Water-gas reaction:
$C + H_2O\ (g) \rightarrow CO + H_2$ -131kJ/mol (4)
Steam reforming:
$CH_4 + H_2O\ (g) \rightarrow CO + 3\,H_2$ -206kJ/mol (5)

In addition to operating condition such as temperature and gasification agent composition and concentration, the diversity on the chemical composition of lignocellulosic materials appears to have a strong impact on conversion techniques employed to produce energy such as pyrolysis and gasification [12]. Moreover, there are inorganic matter that have a catalytic effect on the gasification reactions as Na, K and other have an inhibitory effect such as Si, P and Al [13,14].

The main components of lignocellulosic biomass are cellulose, hemicellulose and lignin with contents changing depending on the biomass type [15]. It usually consists, by weight: 35-50% cellulose, 20-35% hemicelluloses and 10-25% lignin [16,17]. Cellulose is carbohydrate polymer builds of several monomeric D-glucopyranose; from hundreds to over 10000; which covalently linked via β-1,4 bonds forming a linear chain [13,18–20].Contrary to cellulose, hemicellulose has a complex amorphous structure of various pentose and hexose polymers located in the cell wall association with lignin[21,22]. Lignin can be considered as the natural polymer composed of a group of three phenol-based structures with a higher energy density than cellulose and hemicellulose[16,21,23]. The analysis of cellulose, hemicellulose and lignin using FTIR test was done by H. Yang et al [17]. He reported that the chemical groups OH and CO in cellulose has presented the highest IR absorbance. Hemicelluloses has the largest amount of C=O compounds while lignin was characterized by its abundance on the chemical groups $-O-CH_3$, C-O-C and C=C.

It is important to note that there are positive challenges associated with properties of cellulose, hemicellulose and lignin that have motivated many researches based on these main components using different way such as thermogravimetric analysis TGA, different gasifying agents, temperatures, catalysts and different gasifiers [21,24–30].

This work aims to optimize the air–gasification process of the biomass lignocellulosic Cistus monspeliensis (CM) and major lignocellulosic compounds under different gasification conditions in semi-batch reactor. The data obtained provided basic knowledge for the development of products derived from the gasification of cellulose, hemicellulose and lignin which help in the design of devices and the validation of computational models, while CM gasification presented unprecedented results.

2. Materials and methods

2.1. Pure biomass

The pure biomass CM was obtained from nature. It was crushed and sieved to a size of 200 µm to avoid heat and mass transfer limitations. It was then dried in oven at 120 °C for 12 h in order to remove the moisture and finally, enclosed in well-sealed bottles.

2.2. Bio-polymers

The three macromolecular cellulose, hemicellulose and lignin were used in their pure form. cellulose (Ref. 1.02330.0500-500G) was purchased from Merck, hemicellulose (Ref: X0078-100G) was obtained from Chemical Company Co and lignin (Ref: 471003-100G) was purchased from Sigma-Aldrich.

2.3. Semi-batch reactor

The gasification of the different samples was carried out in a semi-batch reactor, shown in figure 1. The reactor consisted of a quartz tube (Ø = 90 mm, L = 970 mm) equipped with a stainless steel spoon. Nitrogen of 200 $mL.min^{-1}$ flow was used as a tracer gas for all experiments. Air fraction varies between 0 and 48% of nitrogen to properly examine the gasification of the air.

The reactor was externally heated by a 1200 W electrical furnace up to the desired temperature between 750 and 900 °C. In each experiment, 0.5 g of the sample was placed in the spoon kept at the mouth of the reactor and the latter was closed tightly. When the temperature was reached, the spoon was moved to the middle of the reactor, where the reaction took place. After 10 minutes, heating was stopped and the reactor was left to cool to room temperature. A cooling system was connected the exit of reactor. This system keeps the temperature -10. The gases are trapped in sample bags for analysis by gas chromatography (GC).

In all gasification experiments, two tests for each run were performed to investigate the repeatability. As well as the gasification time was set at 10 minutes after the assurance that there was no gas production after 10 min.

2.4. Gas product analysis

The "Perkin Elmer GC Clarus580" chromatograph is used to analyze gases by gas chromatography; it is equipped with two detectors: a flame ionization detector (FID) detects hydrocarbons as well as CO and CO2 and a thermal conductivity detector (TCD) can identify non-carbon components such as H_2 and N_2. The instrument also includes a Shincarbon St 100 120 column, a methanizer and a hydrogen generator. The oven temperature is set between 100 and 200 ° C. The hydrogen generator provides a stable source of hydrogen to keep the flame of the FID burning. The carrier gas used here is argon.

3. Results and discussions

3.1. Optimization of biomass CM Gasification

3.1.1. Effect of air fraction and temperature on the gas product yields

The effect of air fraction on the gas yields was experimentally studied by varying its fraction in nitrogen between 0 and 48%. Since temperature is considered as the key of the gasification process, the reaction temperature was varied from 750 to 900 °C for each air fraction during the experiments.

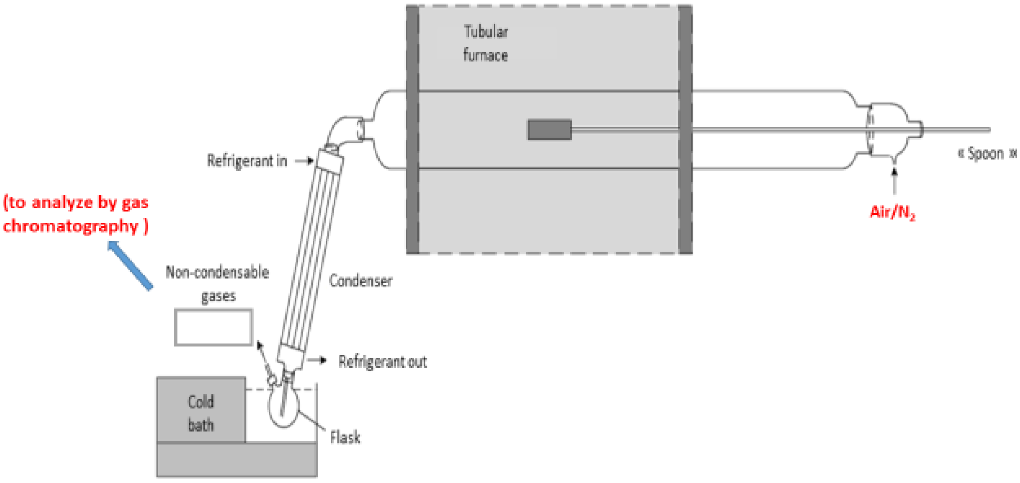

Figure 1. Schematic diagram of semi-batch reactor

The gas yields obtained from the gasification of CM biomass are shown in Table 1.

The differences in the both temperature and air fraction resulted a crucial effect on the gas yields. The evolution of gas in yields were as follows: from 37.82 to 52% at 0% air, from 47.39 to 56.23% at 4% air, from 57.69 to 68.14% at 8% air and from 60.56 to 82.04% at 12% air. It can be concluded that higher temperature bringed to higher gas yield and favored its production compared to char and tar, similar results have been reported in the literature on different biomasses material [5,31–36].

Table 1: Gas yields of CM biomass at different temperature and different air faction

Temperature (°C) / Air-fraction (%)	750	800	850	900
0	37.82	41.92	46.08	52.31
4	47.39	48.64	56.82	56.23
8	57.69	63.42	67.47	68.14
12	60.56	72.37	81.42	82.04

At elevated temperature, the largest gas production can be obtained during initial pyrolysis step. The increase in gas yield is due to endothermic reactions, tar cracking reactions and char gasification reactions, which are favorable at higher temperatures [5,36,37].

The influence of the air fraction and higher temperature generated more energy for gasification and increased the carbon conversion efficiency [38].

3.1.2. Effect of air fraction and temperature on the Gas compositions

It is obvious that combustible components determine the quality of gas and energy efficiency. That is why the effect of air fraction and temperature in gas composition for CM gasification was studied. The gas chromatography analysis showed that CO, H_2 and CH_4 were the permanent gas.

For the resulting plot, see figure2. In the different cases of gasification, the fractions of CO and H_2 showed a marked increase with the increase of temperature.

Whilst the CO_2, CH_4 and C_2H_4decreased. It can be deduct that H_2 and CO yields were improved by higher temperature. This behavior was reported by different researches [31,39–41].

The amount of hydrogen produced at 0% air is highest for all the gasification temperatures: the maximum value of 31.49% was obtained at 900 °C. CO content reaching a maximum of about 49% at 900 °C when air fraction equal to 4%. At lower air fraction, carbon converts to CO instead of CO_2 [37].

High amounts of H_2 and CO emphasized that solid-gas reactions were predominant. The complete decomposition of the large hydrocarbons during the pyrolysis phase caused the formation of char and gas, which reacted completely with the oxygen in the gasification phase. It is also reported that temperature promotes kinetically the endothermic reactions such as reactions (2) and (4) [38,42–44] resulting in increased H_2 and CO contents and decreased CH_4 and CO_2 contents.

Moreover, when the results were judged in relation to the air fraction variation, it was found that the increase of the air fraction decreases the production of CO and H_2 and CH_4.

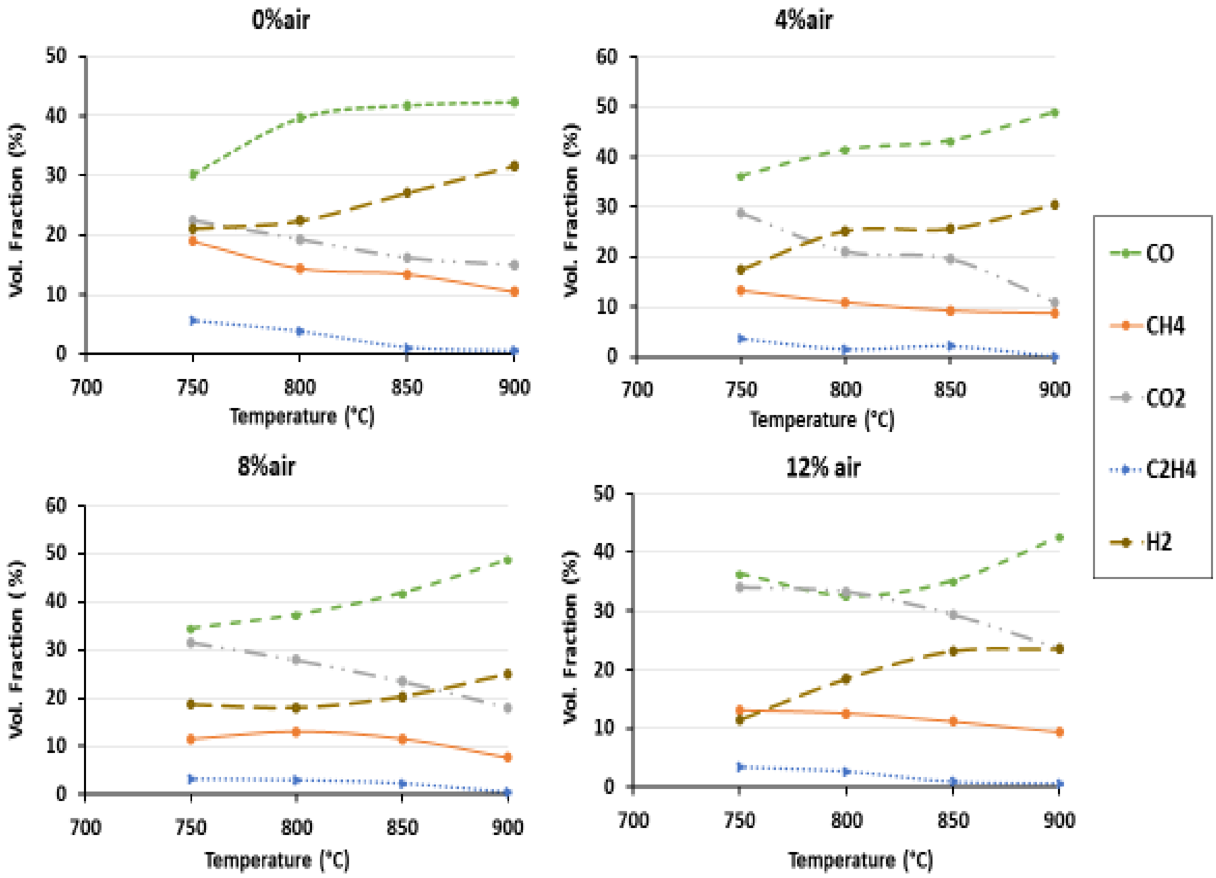

Figure 2. Gas composition of CM biomass at different temperature and different air fraction

3.1.3. Optimization of air fraction on the gasification of biomass CM

The effect of air fraction is well defined in figure 3, which represented the compounds in the gases produced at900 °C and different air fraction: 0%, 4%, 8%, 12%, 24% and 48%.

The CO_2 concentration decreased from 14.97 to 10.74% with increasing air fraction from 0 to 4%. However, the continuous increase of air from 4 to 48% increased the CO_2 concentration to 45.26% and decreased the CO, CH_4 and H_2. Those observations reveals that the presence of excess air enhances the char oxidation than char-gasification and leads to a complete combustion of H_2, CH_4, CO and gaseous hydrocarbons from pyrolysis, which resulted in intense increases in the CO_2 amount and farther decreases in H_2 and CO [38,4].

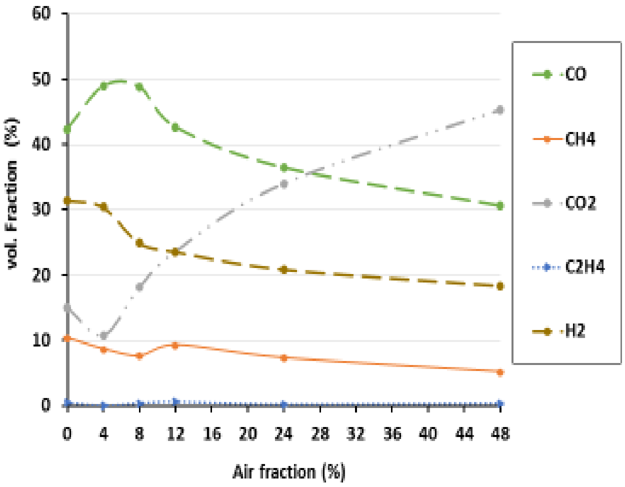

Figure 3. Gas composition of CM at 900 °C and different air fraction

On the one hand, the existence of adequate air fraction in the system could strengthen the gasification, so improving the carbon conversion. On the other hand, more air in the system leads to more CO_2 production, and increases the content of unwanted components [38].

4% of air was found to be the optimal fraction in the following experiments.

3.2. Optimization of cellulose, hemicelluloses and lignin Gasification

3.2.1. Effect of air fraction and temperature on gas product yields

The gas yields from gasification of cellulose, hemicellulose and lignin in the temperature ranging of 750–900 °Care reported in Table 2. The experiments were carried out into gasification cases: 0 and 4% of air fraction. Similar to the results in Table 1, increase in temperature and air fraction has a positive role on the gas production for all samples.

The percentages of total gas produced at the maximum (900 °C) for cellulose, hemicellulose and lignin could reach 60.46%, 50.2% and 40.45%, respectively in the case of 0% air. However, those percentage increases with introduction of air (4%) in the gasifier to 85.38%, 65.28% and 55.63% for the three samples, respectively.

Cellulose and hemicelluloses showed higher total gas production rates than lignin in all results. Whiles the higher yield of char and the low liquid yield were produced by lignin gasification. The data is in agreement with different works: Rutkowski [45] reported that the pyrolysis of cellulose generates a low yield of residue (34 wt%) relative to that of xylan (42wt%) and lignin (51wt%). A highest yield (55.3wt%) of cellulose of gas has been reported by Wu and al [32] during pyrolysis/ /gasification in the absence of catalyst. While the gas yield of xylan and lignin was (44.42wt%) and (21.5wt%), respectively. C. Quan et al [26] compared in his work the pyrolysis product distribution of the three main component. He found that the pyrolysis of lignin gives a remarkably high yield of char (61%) and the lowest yield of tars (0.5%). Those observation that the degradation of cellulose resulted high content of volatiles and the lowest one was produced from lignin degradation were also according to the experimental results obtained with TGA [17,45].

The various behaviors observed can come from the inherent structure difference and the chemical nature of the three samples[26]. Lignin contains relatively less volatile compounds and only lost about 50% of its mass on a carbon basis during volatilization [25,28–30]. Its chemical structure characterizes by the difficult decomposition that gives it a high thermal stability leads to high residue yield [26,32].

In addition, TGA-DSC analysis of hemicellulose and lignin has shown the existence of exothermic reaction [17], these reaction favors the carbonization process [48] leading to a high yield of solid residues for the hemicelluloses and lignin decomposition. For cellulose decomposition, only the endothermic reaction were observed [17], and therefore produced the greatest amount of volatile contents.

Table 2: Gas yields of cellulose, hemicellulose and ligninat different temperature and different air faction

	Temperature (°C) / Air fraction (%)	750	800	850	900
Cellulose	0	51.32	55.52	55.98	60.46
	4	81.49	81.92	82.39	85.38
Hemicellulose	0	48.82	51.63	54.58	50.2
	4	60.84	67.97	69.62	65.28
Lignin	0	36	38.92	39.76	40.45
	4	47	53.69	63.62	55.63

3.2.2. Product gas composition

The influence of temperature on gas composition of biomass compounds was studied in the both case o% and 4% air. The results are showed in figure 4.

During the gasification of cellulose and hemicellulose, the CO production is important but the temperature does not have a significant effect on the evolved volume fraction. Except at 850°C, there was a small changing in hemicellulose behavior, CO amount showed an optimum of 48.25% in pyrolysis step, while, in the case of gasification, the amount of CO decreased to 36.28%. Like in CM gasification, CO_2 production decreased by rising in temperature.

At 0% air, the CO concentrations was in the order of cellulose (48.04-51.79%) > hemicellulose (42.40-42.79%) > lignin (16.68-11.31%). While those of CO_2 was as follows: hemicellulose (16.37-14.80%) > lignin (16.68-11.31%) > cellulose (12.63-8.20%).

Cellulose exhibited the higher CO amount and the lowest CO_2 one, while hemicellulose and lignin display the large amount of CO_2. Similar data were obtained by S. Wang et al [48] when they studied the interaction of biomass components during pyrolysis at a temperature of 540 °C. Carole Couhert [49] found that flash-pyrolysis of

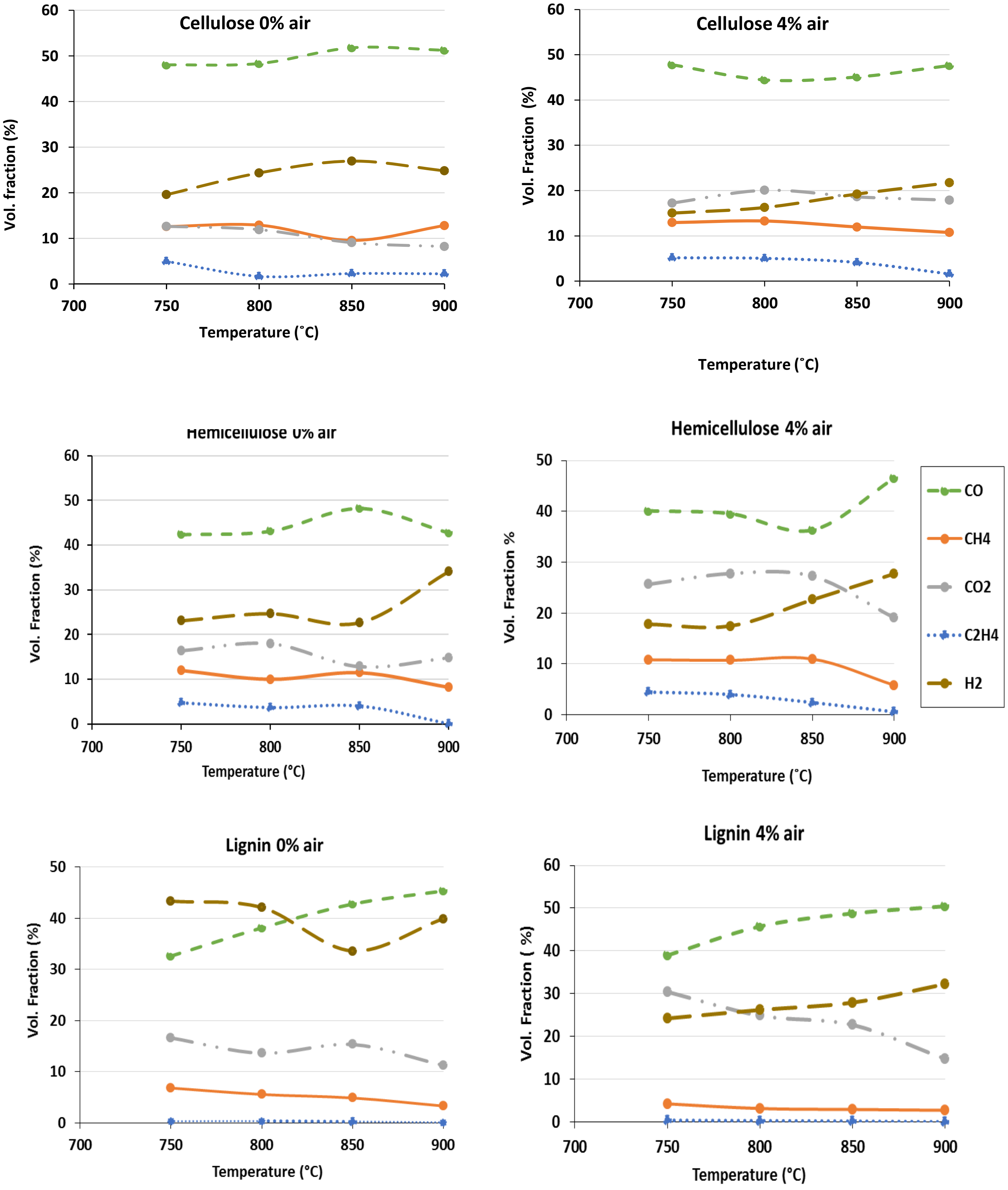

Figure 4. Gas composition of cellulose, hemicellulose and lignin at different temperature and different air fraction

xylan at 950 °C resulted the highest amount of CO_2 compared to cellulose and lignin.

In regards to FTIR analysis of cellulose, the presence of large amounts of carbonyl chemical groups (C-O-C) in its composition could favor the production of carbon monoxide during the destruction [17], [32]. Cellulose may also have produced more CO due to secondary reactions of primary volatiles and cleavage of aldehyde group (R-CHO). In addition, the abundant presence of chemical groups in hemicellulose is suggested to the production of carbon dioxide [50,51].

The presence of 4% air in the process causing less diminution of CO concentrations obtained from cellulose and hemicellulose, it takes the same trend of CO obtained from CM gasification. In contrast, CO product from lignin gasification increased with temperature reaching up to 50.43% of the gas composition at 900°C. Probably this lignin behavior due to that the activity of the chemical bonds in lignin covered an extremely wide range, which led to the degradation of lignin occurring in a wide temperature range [52]. Moreover, component contents affected gasification strongly by changing char structure, and that the char from lignin had higher porosity and reactivity [53].

Meanwhile, H_2 concentrations increased substantially from 19.58 to 26.95% at 0% air and from 15.02 to 21.73 at 4% air for cellulose; from 23.10 to 34.09% and from 17.09% to 27.73% for hemicellulose. The results indicated that lignin produced more H_2 than others. These results were in good agreement with the finding reported by other researchers [26,27]. It might be attributed to the higher content of aromatic ring and O-CH_3 functional groups in the lignin samples, as H_2 is mainly from the cracking and deformation of C=C and C-H groups [17,26]. The maximum production amount of H_2 was 43.34%occurs at 750°C and 0% air, which is very close to 800°C. Similar to H_2 behavior in CM gasification, presence of air causing less amount of H_2 compared to that at 0% air for the three samples.

CH_4 yields obtained from lignin were less than there obtained from cellulose and hemicellulose. The same observation have been showed by Tian et al [27]. For lignin, CH_4 decreased slightly with increasing temperature in the tow gasifying media. While, for cellulose and hemicellulose, CH_4 yield did not showed a significant changes when temperature and air fraction increased. CH_4 is mainly brought by the cracking of methoxyle [17].

4. Conclusion

Gasification is a green process and it has proven that the energy recovery of CM and pure woody pseudo-components is very important. The variation in operating conditions of gasification experiments leads to the following important conclusions:

The increase of gasification temperature and air fraction was found to increase the production of gas during the pyrolysis/gasification process of biomass, cellulose, hemicelluloses and lignin.

In each run, the main components of the gas phase were CO, CO_2, H_2, and CH_4.

During pyrolysis and gasification of CM, the increasing of the temperature leads to the significantly increase of the CO and H_2 contents reaching a maximum value of about 50% and 31.49%, respectively, and the decrease of the CH_4 and CO_2 contents. While the increasing of air fraction from 0% to 48% in the gasifying media reduced the CO and H_2 contents in gas and increased the CO_2 concentration. 4% of air was found to be the optimal value in the gasification of CM and the main components of biomass.

Gas components products from the gasification of cellulose, hemicellulose and lignin are different to each other. The CO pyrolysis yields was in the order of cellulose (48.04-51.79%) > hemicellulose (48.04-51.79%) > lignin (32.64-45.34%).

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