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Study of the reactivity of Casamance vegetable and agricultural waste char gasification with steam and CO₂

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The increasing energy demand coupled with the need to reduce greenhouse gas emissions and the threat of exhaustion of oil reserves make us consider a possible recourse to the use of biomass waste as a source of renewable energy. Nowadays, gasification is not yet economically and operationally attractive for the power industry and more research is needed to facilitate the process and improve the desirability of the gasification process. Gasification tests were conducted on five wastes char mainly of agro-sylvo-pastoral residues, in order to study the behaviors of char conversion based on experimental data. Peanut shells, palm shells, cashews nut shells, cashew wood and “kaicedrat” wood char obtained by pyrolysis at 450°C are used. The samples were gasified at three different reaction temperatures (950 to 1050°C) in a fixed bed reactor, using steam or CO₂ as gasification agent and with average fraction of particle size 630 and 3000 µm. The experimental parameters, which affect the char’s reactivity, are reviewed similarly to those related to the char and its structural features and operation parameters. Gasification kinetic conversion was studied at different models: the volume reaction model (VRM) and shrinking core model (SCM) in order to interpret the char conversion data. Further, the activation energy and pre-exponential factor were determined using the Arrhenius correlation. The experimental results showed that more syngas ((CO + H₂)) of high quality were obtained at 1000 to 1050°C during char gasification with steam or CO₂. The present results showed that temperature has a positive effect on kinetic char conversion. In addition, the low heating values obtained as a function of temperature depend on the nature of sample. For further investigation, it can be shown that the reaction rate is dependent on the char samples. Thus comparing the five biomasses, particular importance about reactivity and lower heating value (LHV) is attached to cashew nut shells, palm shells and peanut shells.

Key words: Kinetic of char conversion, char samples, char-CO₂ gasification, char-steam gasification, lower heating value (LHV).

INTRODUCTION

Gasification processes are currently receiving attention in terms of a cleaner and more energy efficient char

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conversion technology. Several methods were being studied in order to develop more efficient and environmentally friendly char conversion technologies. Thus, coal compared to biomass's char is the main feedstock used in gasification processes.

Senegal being an agricultural based country with a corresponding large supply of biomass resources: peanut shells, sorghum, palm shells, cashew nut shells, wood residues and cereal stems. These resources can be used for energy purposes (combustion and gasification). Pyrolysis occurs on the order of short time after injection of pulverized broken chars to volatile matter into the bed. Gasification includes heterogeneous reactions between char and gases reactions. Thus, Liu et al. (2015) stipulate that water and carbon dioxide are two regular gasification agents and water has been widely used as a gasification agent, but is recently limited to reach in the energy development sector and alternatively, using CO₂ as a gasification agent has been given attention recently. It has been speculated that understanding the kinetics of the CO₂ char gasification under elevated temperature and pressure is helpful to better organize many industry processes. Biomass coal, however, have different chemical and physical properties, that is, volatile matter, ash composition, density, calorific value, H/C and O/C molar ratio. These differences in the properties lead to different reactivity and thermal characteristics during co-processing.

So, different studies have been performed on char gasification in fixed beds. Nowadays, gasification is not yet economically and operationally attractive in the energy industry and more research is needed to facilitate the process and improve the desirability of the gasification process (Karimipour et al., 2013). In addition, the complexity of gasification process, the differences in the gasifier, physical properties of waste biomass, and operation parameters have strong influence on the mass and heat transfer between char of biomass during gasification. Comparing and analyzing gasification processes, it is useful to examine performance parameters: such as temperature, particles size, and types of char. Char-CO₂ reaction and char-H₂O reaction are both fundamentally important reactions (Li et al., 2017; Lin and Strand, 2013). The mechanisms of the char-CO₂ reaction and char-H₂O reaction, the effect of particle size and experimental temperature have been studied extensively by many authors, and they have equally worked on the char gasification mechanism in CO₂ or H₂O (Coetzee et al., 2013; Mani et al., 2011; Hattingh et al., 2011; Wang et al., 2009).

Several parameters have a direct effect on the mechanism of gasification: the temperature is the most important parameter, because its effect on others parameters such as the char kinetic conversion, and the calorific value. The data variations of conversion rates of carbon, which depend on the time of char samples gasification under steam atmosphere or under CO₂ atmosphere, were examined to clarify the factors that

control the mechanism of gasification (Huo et al., 2014).

There have been numbers of theoretical or mathematical models, which can explain behaviors of carbon kinetic conversion against time or rate variation. The kinetics of the CO₂ and steam gasification of biomass chars, were studied by several researchers using various kinetic models, as summarized by Everson et al. (2006), Guizani et al. (2013) and Yang et al. (2017). Chen et al. (2013) studied the effect of pyrolysis conditions on the char gasification with mixtures of CO₂ and H₂O using the shrinking core model (SCM) to derive reaction rate constants. Jeong et al. (2014) studied the go-gasification of coal-biomass blended char with CO₂ at temperatures of 900 to 1100°C using SCM and volume reaction model (VRM). Various methods were being studied in order to enhance more efficiently and environmentally friendly char conversion technologies as reported by Coetzee et al. (2013). A number of research studies have been recently carried out on the gasification of charcoal, peanut shells, cashew nut shell, biomass coal, coal of shell of oil palm and coconut shells, sorghum stalks and cotton stalks, focusing mainly on gasification efficiency of the system.

Extensive studies on methods of biomass gasification had also been conducted on lower heating value (LHV) of gas (Xie et al., 2012), obtained in the syngas production by two-stage method of biomass catalytic pyrolysis and gasification a LHV (10.80-14.75 MJ/Nm³). Almeida et al. (2017) studied steam gasification of rice husk in a fluidized bed reactor and generated syngas with a LHV of 11.18 MJ/Nm³ at 750°C. So Kreckkaiwan et al. (2013) in the synergistic effects during co-pyrolysis/gasification of biomass and sub-bituminous coal, reported that the apparent contradictions of the results found in the literature can be function of experimental parameters used from one author to other, such the temperature, pressure, heating rate, the type of coal or biomass, and the origin of the material used. Thus, this study aims to investigate the effects of temperature and particle size on the low calorific value, and to study the effect of char samples (char of peanut shells, palm shells, cashews nut shells, cashew wood and "kaicedrat" wood) on their kinetics of conversion, evolution during the gasification with steam and CO₂. Gasification reactivity rate was also studied at different temperatures and with two atmospheres using the volume reaction model (VRM) and shrinking core model (SCM), in order to interpret the carbon conversion data. This kinetic comparison study of the effect of these Casamance samples on char kinetic has never been studied in the literature.

MATERIALS AND METHODS

Experimental samples

Five chars of biomass (peanut shells char (C. peanut), palm shells char (C.palm), cashews nut shells char (C. cashew), cashew wood

Table 1. Elementary and proximate analysis of the different char of samples used.

Biomass	Palm Shell	Cashew N. Shell	Peanut Shell	Wood (mean)
Proximate analysis (Wt.%)				
FC	13.4	17.3	19.6	17.4
VM	84.9	81.8	65.4	82.1
Moisture	3.0	6.1	9.3	0.21
Ash	1.2	0.9	5.7	0.12
LHV	21.2	21.4	17.0	18.6
Elementary analysis (Wt.%)				
Carbon	49.5	48.7	48.1	51.6
Hydrogen	6.00	7.0	5.5	6.3
Oxygen	43.6	43.9	30.0	41.5
Nitrogen	2.1	0.4	1.3	0.4
Soufre	0.16	0.1	0.08	0.1

char (C.cashew wood) and “kaicedrat” wood char (C.kaicedrat wood)) were used in this study. The samples are typical agricultural and vegetal residues from south natural region in Senegal (Casamance). In addition, after the abundance and good energy countenance of these residues, the following motivated us to use them:

- (1) The total absence of a scientific presentation of these Casamance residues in the field of thermo-chemical conversion,
- (2) Limitation of biomass open air burning and changing to energy resource used.

In order to eliminate the effect of moisture content prior to each test samples, they were dried at 105°C for several hours (24 h) and then stored in plastic bags to prevent extra absorption of moisture from atmosphere before the pyrolysis. The pyrolysis experiment was carried out under a muffle oven. With 100 g per test and per the sample used: within an approximate test duration of 15 min for peanut shell, and 20 min for palm shell, cashew wood, “kaicedrat” wood and cashew nut shell. The pyrolysis temperature is fixed at 450°C under inert atmosphere. All char samples obtained were ground and sieved into two fractions of particles size <630 and 3000 µm. The properties of the samples used are shown in Table 1. This analysis gives an idea of the quality of energy gross provided by each biomass. The mass proportion of fixed carbon (FC) and volatile matter (VM) are two factors that indicate the amount of stored chemical energy by the biomass. More the MV/CF ratio is high and more energy capacity of this biomass is important. Also, Nipattummakul et al. (2012) announced in the study of gasification of palm waste, mangrove wood and waste paper that the difference of evolution of syngas production is due to the difference of the volatile matter content between samples.

Experiment description

The sample char gasification tests were conducted using a fixed bed reactor (36 mm internal diameter and 350 mm height) equipped with a porous plate for bed support. Figure 1 shows a flow diagram of the fixed bed reactor. The main elements of this fixed bed reactor diagram consist of three sets: gas analyzer, gas condensation and cleaning system.

After reactor has been preheated, 15 g of char is mixed with 70 g of sand and charged in the reactor. This char sample was carried

out within a nitrogen atmosphere, until the desired temperature. Sand was used in order to improve heat transfer inside bed particles and for minimizing the preferential gas passage. The reactor temperature is controlled by means of a thermocouple (TC), in contact with the sample bed and connected to a temperature controller. The gasification tests were carried out isothermally at 950, 1000 and 1050°C, using steam (H₂O) and CO₂ (90%) and carried in an inert flow of 10% of nitrogen (N₂). Flow rates of CO₂ and N₂ were fixed by the use of mass flow controllers while the water flow rate was adjusted by a piston pump (made 510 water pump).

Before entering the reactor, the reactive N₂, CO₂, and H₂O cross a preheating section. The composition of the produced gas is obtained by online gas analysis, using an SRA-Instruments gas analyzer (µGC), after gas condensation and cleaning.

The experimental data obtained from the µGC have been processed and presented subsequently.

RESULTS AND DISCUSSION

Char-CO₂ and char-steam gasification experiments for different particle sizes of five chars and three temperatures of gasification were all investigated. The carbon conversion is obtained from the following equation:

$$X_i = \frac{m_0 - m_t}{m_0 - m_{ash}} \quad (1)$$

where m_0 is the initial mass, m_t is the mass of the sample at time t , and m_{ash} is the mass of ash remaining in the reactor.

The effect of the main operation variables such as temperature, char size particle and char sample was studied, by evaluating and comparing the char carbon conversion or kinetics models.

The carbon conversion, X (Equation 1) was defined as the total carbon contained in the produced gas (CO, H₂O and CH₄), with respect to the total carbon contained in

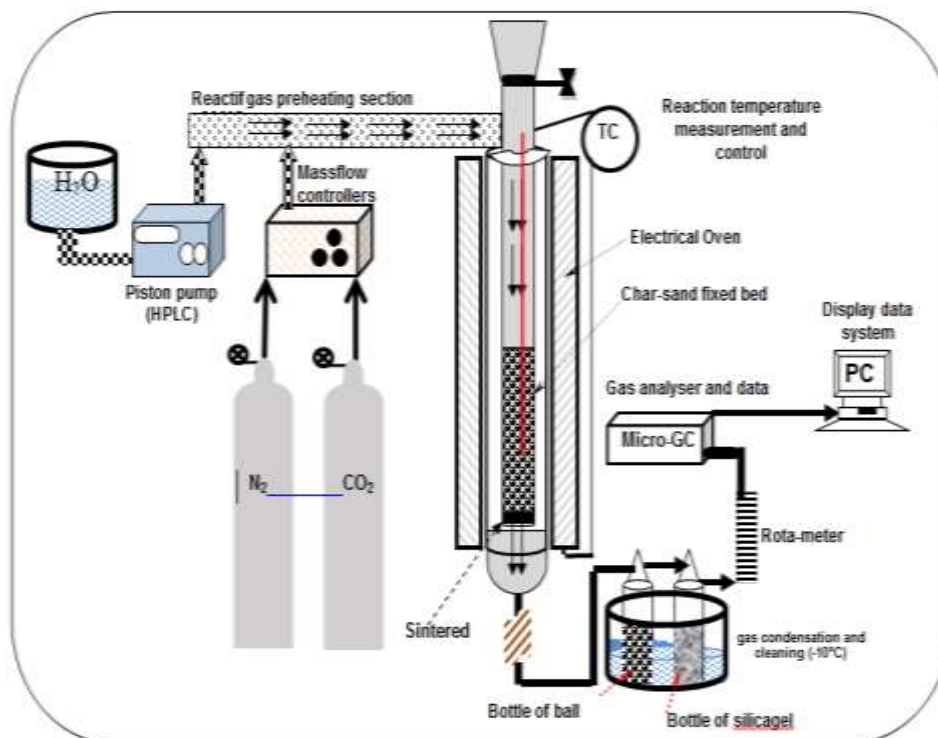


Figure 1. Simplified representation of the fixed bed reactor's system.

the char fixed bed. The amount of gas generated during gasification tests was calculated from nitrogen balance, since the amount of nitrogen fed in and the composition of nitrogen evolved are known.

Effect of temperature on the rate of carbon conversion (steam or CO₂)

The effect of temperature on kinetic of char conversion during gasification with steam and CO₂, with five char samples is as shown in Figure 2. Using the experimental data of syngas, the evolution of X (rate of carbon conversion) as a function of time for various temperatures, was plotted for all the samples (Figure 2).

The influence of gasification temperature on conversion rates is very important, since all of thermo-chemical steps of the char-CO₂ and char-steam reactions for syngas production are temperature dependent (Almeida et al., 2017). The amount of volatile matter, which is cracked from the solid, is a function of temperature. It has been shown by several authors that higher temperatures favor the production of the gas syngas (Xie et al., 2012; Kuprianov and Arromdee, 2013). Thus, the result example of temperature effect on rate conversion is as shown in Figure 2. The curve in Figure 2 shows that the char are sensitive to temperature variations, where an increase in temperature results in an enhancement in reactivity of carbon. This effect can be explained by the

principle of Le Chatelier: that the products formed during the endothermic reaction are favored at high temperature. This trend was also observed on two particle sizes of the sample. The present results are in agreement with Coetzee et al. (2013), Xie et al. (2012), Zhang et al. (2017), Wilk and Hofbauer (2013), and Skodras et al. (2015), who have also noticed that the temperature has a positive effect on the conversion of char. A rise of temperature favors chars conversion and gasification reactions. This result supports the choice of kinetic based on activation energy used (Arrhenius equation).

The models can be used to predict the conversion of the biomass char gasification and optimize the design and operation of the gasified (Zhai et al., 2017).

Comparison of char reactivity

Kinetic parameters

The study of kinetic gasification of the char of all samples was conducted using the volume reaction model (VRM). This model was applied for interpreting experimental data. The kinetics parameter such as the activation energy and the pre-exponential factor with steam and CO₂ was determined using the Arrhenius Equation 2 or 3.

$$k = k_0 \exp\left(-\frac{E_a}{RT}\right) \quad (2)$$

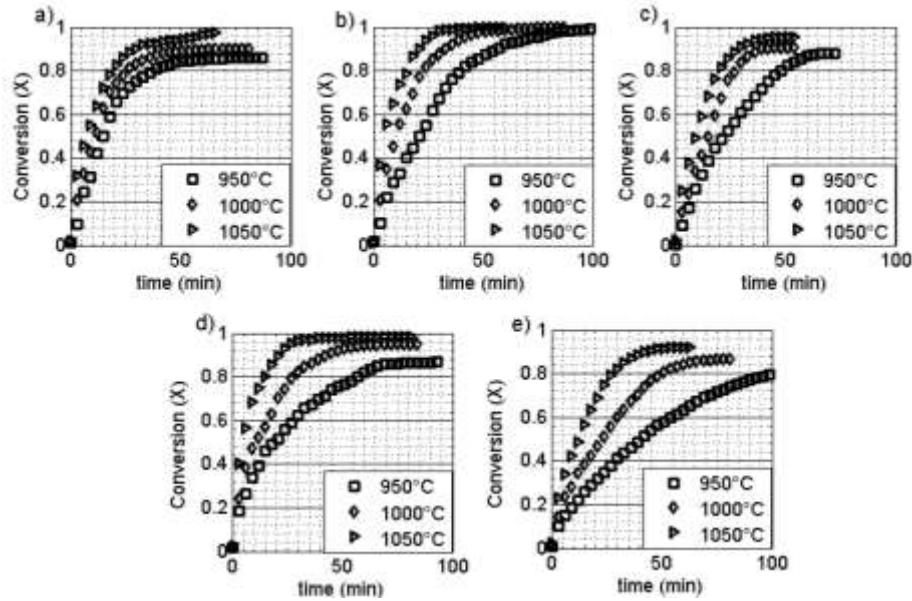


Figure 2. Effect of the temperature on the X of the various char of C.peanut (a), C.cashew wood (b), C.kaicedrat (c), C.cashew (d), and C.palm (e) under steam atmosphere.

Equation 2 can be further transformed as

$$-\ln(k) = -\ln(k_0) + \frac{E_a}{RT} \quad (3)$$

where k_0 , E_a , R , and T are the pre-exponential, activation energy, universal gas constant, and the experimental temperature, respectively.

This equation represents the physical and chemical profiles of the sample and it is represented by volume reaction model or shrinking core model.

Therefore, this kinetic model, which is based on the assumption that gasification takes place homogeneously (Skodras et al., 2015), seems to describe the char samples gasification with CO_2 and with steam quite efficiently.

The reactivity of coal gasification can be represented through VRM model, this model was applied to interpret the coal reactivity and is shown in Equation 4.

$$\frac{dX}{dt} = k_{VRM}(1-X) \quad (4)$$

where (dt) is the variation of residence time of the char conversion.

The shrinking core model (SCM) considers that the gasifying agents react on the surface of nonporous grains or in pore surfaces within the solid (Zhang et al., 2008). According to different assumptions, the reaction rates in the chemical control regime can be expressed as:

$$\frac{dX}{dt} = k_{SCM}(1-X)^{2/3} \quad (5)$$

To study the applicability of the selected kinetic models and to predict the kinetic behavior of the studied high ash coal samples, the models were fitted with the experimental data ($X=0$ to $X=0.5$). For this operation, the reactivity should refer to a specific conversion degree. Reactivities at 0% or 50% of char conversion are often used for the determination of the kinetic parameters; the latter value is actually the most commonly selected parameter in several similar investigations (Xiao and Yang, 2016).

Samples were tested in the same experimental conditions (the same temperature, reactional atmosphere and size). The results obtained from the VRM and SCM of these samples are as shown in Figure 3.

Figure 3 shows the gasification trends for C.cashew wood, C.kaicedrat wood, C.cashew, C.peanut, and C.palm samples under CO_2 atmosphere at 950, 1000, and 1050°C. The results of Arrhenius equation are plotted in Figure 3; however, the pre-exponential factor and the activation energy obtained from these plots, for each test conditions, are listed in Table 2.

It can be noticed from Figure 3 that the activation energy (E_a) is obtained from $-E_a/R$ from cashew wood $< E_a$ of kacedrat wood $< E_a$ of cashew shell $< E_a$ of peanut shell $< E_a$ of palm shell (Figure 3). Thus, when comparing the kinetics of char conversion, it can be noticed that the char kinetic conversin follows the order: C.cashew wood (fastest) $>$ C.kaicedrat wood $>$ C.cashew shell $>$ C.peanut

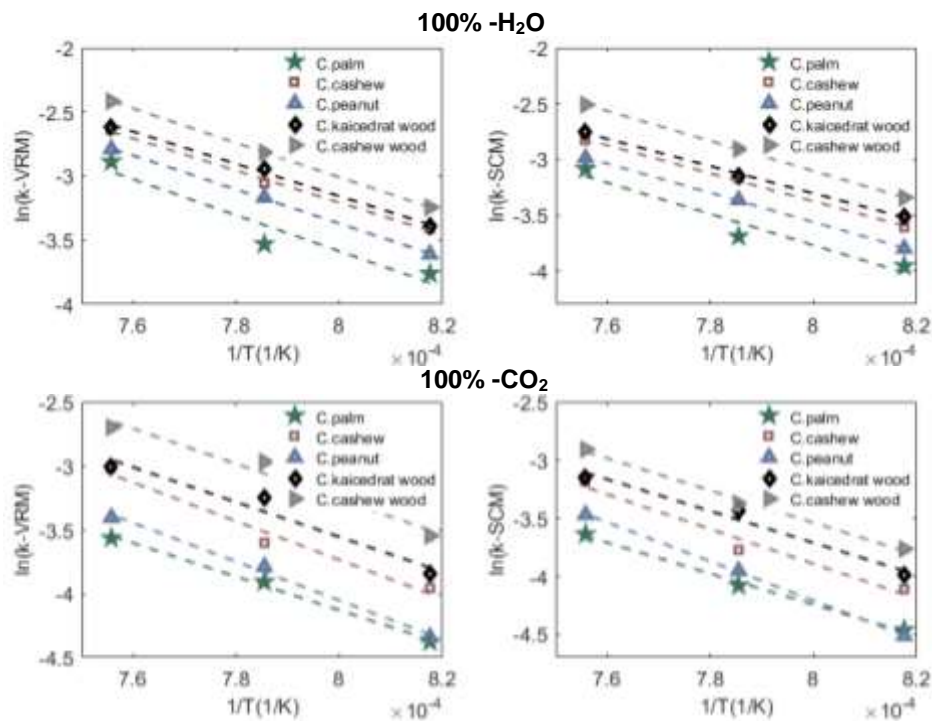


Figure 3. Arrhenius plots of sample char gasification reaction with various char sample.

> C.palm (slowest).

This general trend showed several interpretations for all the different experimental conditions. These effects of the experimental conditions on char reactivity can be also explained by the compositions determined from a proximate analysis of Table 1. However, Zhang et al. (2008) and Ding et al. (2014) showed that the chemical elements (K and ash for examples) are responsible of the effect on the type of char samples considering the reactivity of char conversion during gasification, and they gave several correlations between them (these elements) and interpretations. Nevertheless, based on an analysis of the literature review on ligno-cellulosic chars, Blasi (2009) concluded that the nature of the lingo-cellulosic biomass has no significant effect on the char reactivity and the differences among various samples can be attributed mainly to the amount and composition of ashes. It can be concluded that, these differences in the overall gasification kinetic of char samples are significant for experimental process. They showed that, depending on the type of biomass, the gasification time will vary. Since the increase of temperature favors the reduction of the ash content of the biomass, then the present results are in agreement with Blasi (2009) conclusions. This effect of the char samples on the kinetics could be due to the different reasons of these chars composition, structural properties, the surface area and porosity. The characteristics of the char that affected the reaction rate are essentially the structural property, which includes the

surface area and porosity and the intrinsic reactivity depending on the chemistry and catalytic effect of the ash compounds. This may also be due to the char pores as the structure opens, which allows the gasifying reagent greater contact with the char carbon, and which increases the kinetic char conversion.

As shown in Figure 3, the simple volume reaction model predicts the experimental results for the temperature studied quite well. In agreement with previous investigators (Jeong et al., 2014), at gasification temperatures (900-1100°C), this model fitted the experimental data quite well. This table presents the kinetic parameters (E_a and K_0) determined from the data obtained at 950, 1000 and 1050°C, for C.cashew wood, C.kaicedrat wood, C.cashew, C.peanut and C.palm chars with particle size (0.63 mm) and under CO_2 or steam atmosphere, together with high regression coefficients ($R^2 > 0.9$). The activation energy, function of the samples and different models, obtained for the samples char gasification is between 104 and 131 kJ/mol. The shrinking core model using the parameters describes the present experimental data more than volume reaction model.

Effect of temperature and particle size on the lower heating value of the gas (LHV)

In order to highlight the effect of temperature and the particle size on the LHV gases, the following correlation

Table 2. Kinetic parameters of char gasification.

Sample	Reagent	Models	E _a (kJ/mol)	K ₀ (min ⁻¹)×E ⁺³	R ²
C.Peanut shell			112.91	1.40	0.973
C.cashew wood			110.43	3.36	0.994
C.kaicedrat wood	CO ₂	SCM	110.92	0.62	0.996
C.palm shell			124.44	3.20	0.968
C.Cashew nut shell			111.31	1.89	0.9684
C.Peanut shell			113.70	1.70	0.955
C.cashew wood			108.79	1.67	0.998
C.kaicedrat wood	CO ₂	VRM	109.17	0.585	0.996
C.palm shell			126.00	4.36	0.975
C.Cashew nut shell			112.84	2.39	0.975
C.Peanut shell			109.28	0.94	0.999
C.cashew wood			104.26	2.18	0.999
C.kaicedrat wood	H ₂ O	SCM	106.60	0.65	0.999
C.palm shell			116.07	1.71	0.951
C.Cashew nut shell			107.45	0.78	0.997
C.Peanut shell			110.63	1.29	0.9994
C.cashew wood			103.53	2.24	0.993
C.kaicedrat wood	H ₂ O		104.02	0.90	0.995
C.palm shell		VRM	116.80	2.08	0.919
C.Cashew nut Shell			104.81	1.01	0.995

of Xie et al. (2012) was used:

$$LHV = (30 \times [CO] + 25.7 \times [H_2] + 85.4 \times [CH_4] + 151.3 \times [C_nH_m]) \times \left(\frac{4.2}{1000}\right) MJ / Nm^3 \quad (6)$$

with [CO], [H₂], [CH₄], and [C_nH_m] the molar ratio of CO, H₂, CH₄, and C_nH_m in the produced gas, respectively. According to the Equation 6, high CO, H₂, and CH₄ content of hot reducing gases would be beneficial for the process (Suopajärvi et al., 2013).

Depending on the gasification agent, the method of operation and the process conditions, three product gas qualities can be produced. The calorific values (CV) of the three products gas from biomass gasification are according Suopajärvi et al. (2013): Low CV 4 to 6 MJ/Nm³ (air and steam/air); Medium CV 12 to 18 MJ/Nm³ (oxygen and steam); and High CV 40 MJ/Nm³ (hydrogen and hydrogenation). Gasification products are used in several applications: for example the low calorific value gas can be used directly as a fuel gas in turbines and gas engines (Suopajärvi et al., 2013). It is obvious that the effects of temperature on the gas yield were also related with the LHV (Almeida et al., 2017; Zhang et al., 2017). Figures 4 and 5 show lower heating value (LHV) of the gas at different temperatures and with two particle sizes (630 - 3000 μm). The analysis of the data obtained

reveals that temperature has an effect on the lower heating value of gas (Lv, 2004; Zhang et al., 2017) on their respective studies found that the high heating value of gas product increases when the temperature increases from 850 to 1050°C. It was noted that the differences between the LHV values of gas obtained at different temperatures and different sizes are important.

Regarding Figures 4 and 5, one can observe that the LHV of gas for the small size particles (630 μm) are higher than those from larger particles (3000 μm). It was also noted that the more the temperature is higher the better is the LHV value. It can be concluded that the value of the lower calorific value of the product gas is a function of the temperature and of the particle size. The lower calorific value of the gas is approximately estimated from 9 to 12 MJ/Nm³ for gasification of char with CO₂ and 7 to 12 MJ/Nm³ with the steam. The main gasification LHV gas values obtained is higher than the results obtained by Almeida et al. (2017) and Zhang et al. (2017). The analysis Figures 4 and 5 show that the maximum values were obtained at 1050°C for a particle size of 630 μm reagent char of cashew wood (b), kaicedrat wood (c), peanut shell (a), cashew nut shell (d) and palm shell (e). These results can be explained by the fact that a larger amount of gas was analyzed during char gasification with CO₂ at 1050°C and at a size of 630 μm,

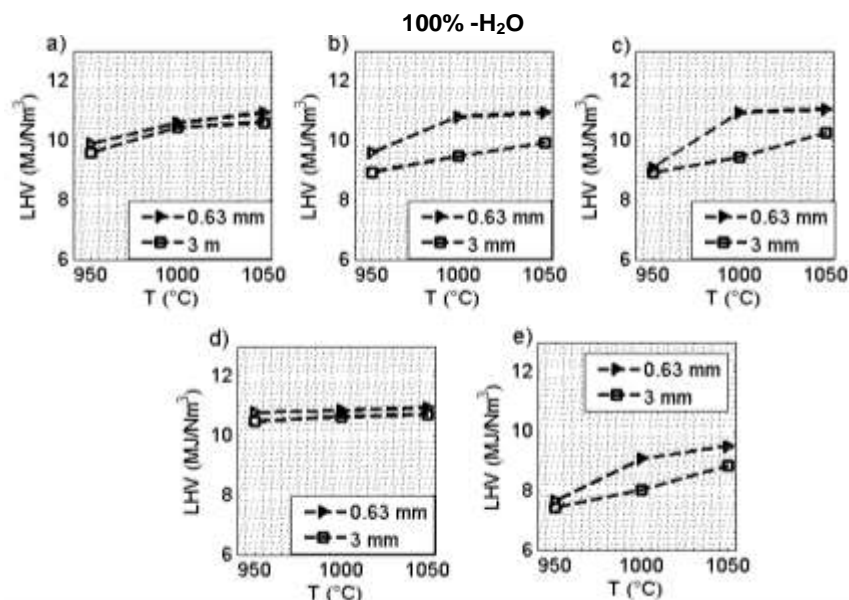


Figure 4. Evolution of LHV of gas versus temperature at different particles sizes under steam.

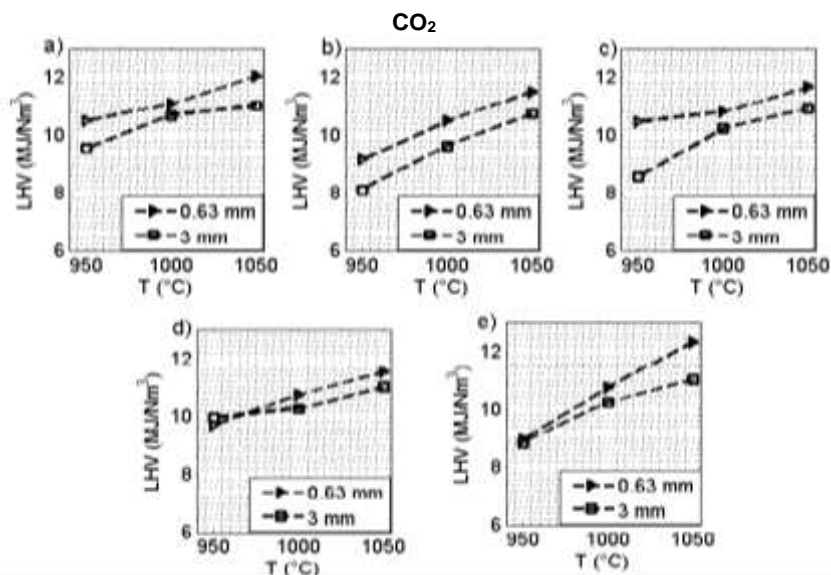


Figure 5. Evolution of LHV of gas versus temperature at different particles sizes under CO₂.

than during the gasification of char with H₂O to 1050°C and a particle size of 630 μm on these samples.

The particular importance will be attached at lower heating value (LHV) to cashew nut shells, palm shells and peanut shells.

Conclusion

In this study, the peanut shells, cashew nut shells, palm

shells, cashew wood and “kaicedrat” wood char gasification with the reacting agents (steam and CO₂) at various temperatures (950 to 1050°C) and for two different particles (630 to 3000 μm) have been applied to investigate the effect of the operating condition.

The results illustrated that, high temperature (1000 and 1050°C) showed an improvement on samples' char conversion for reacting agents. High temperatures improve syngas production, mainly carbon dioxide and

hydrogen, with high carbon conversion rate. The carbon conversion rate obtained is a function of the temperature, due to the gas production during endothermic reactions at higher temperatures. Thus, higher is the temperature (1000 and 1050°C), better is the conversion of char sample during gasification under steam and CO₂. In conclusion, the present results established that the value of the lower calorific value of the product gas is a function of the temperature and of the particle size. The lower calorific value (LHV) of the gas is approximately estimated from 9 to 12 MJ/Nm³ for gasification of char with CO₂ and 7 to 11 MJ/Nm³ with the steam. The maximum values of LHV (~11 to 12 MJ/Nm³) was obtained with a temperature of 1000 and 1050°C, at a particle size of 630 µm during gasification of C.cashew, C.peanut, and C.palm chars under CO₂. The plot of char conversion versus time shows that particle size had significant influence on the char LHV of gas. Among the different particle sizes investigated, at the same temperature, the 630 µm particles size achieved a higher LHV than those from 3000 µm particles sizes. This could be due to the large surface area of fine particle size.

It is also concluded that, the nature of biomass has an effect on the kinetic of conversion and the different behaviours of char during gasification among various samples can be attributed mainly to the amount and composition of ashes.

Finally, reaction kinetic parameters showed the best reactivity on cashew wood char with CO₂, compared to the reactivity of "kaicedrat" wood, cashew shells, peanut shells, and palm shells char with carbon dioxide for the two particles sizes. In view of the results obtained for LHV in this study, the char of the palm shells is 12.32 MJ/Nm³, cashew nut shells is 11.72 MJ/Nm³, and peanut shells is 12.03 MJ/Nm³ with CO₂ or steam at 1050°C and 630 µm. It would be important to continue this study of gasification on the palm shell char, cashew shell char, and peanut shell char, using a gasification study in a mixed atmospheres (100% -H₂O, 75% -H₂O/25% -CO₂, 50% -H₂O/50% -CO₂, 25% -H₂O/75% -CO₂, and 100% -CO₂), of CO₂ and steam, respectively, all carried in a fluid of N₂.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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