Mathematical Model for Estimating Char Production Rates Derived From the Pyrolysis of Rice Hulls and Corn Cobs

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Abstract
There is a need to promote sustainable energy resources that balance consumer fuel demands and minimize environmental impacts. An obvious solution to this problem is renewable energy fuels, such as biomass. Corn cobs and rice hulls are abundant resources for biofuel production in the country of Benin. The objective of this study was to estimate the rate of char production during the pyrolysis phase of the gasification reaction. A mathematical model was developed based on the pyrolysis data at isothermic conditions to predict the char rates of corn cobs and rice hulls at different temperatures. Curve fittings of the data were performed using MATLAB 7.0 software using Newton's method. Predictions from the model confirmed the influence of temperature on the char rates of the examined biomasses, as well as the distinguishable stages observed during pyrolysis. The char yield from rice hulls was greater than from corn cobs regardless of the pyrolysis temperature

Keywords: Biomass - Char – Pyrolysis – Mathematical model.

INTRODUCTION
Biomass-derived fuels include waste from animal and vegetable matter as well as agribusiness sources (Bridgwater 2006). Africa has potential for producing agricultural biomasses. Several studies have been conducted on agricultural biomasses (Keener et al. 1986; Butuk and Morey 1987) to produce energy fuels, which includes ground and whole corn cobs (Keener et al. 1983), shelled corn (Schonauer et al. 1984), cotton gin trash (LePori et al. 1981), and rice hulls (Zhang et al. 2009). Biomass conversion to fuels follows either thermo-chemical or bio-chemical methods (McKendry 2002). Pyrolysis, which is a thermo-chemical conversion method, is an endothermic reaction that converts biomass to a mixture of gases and solid residues, including char and tar. Many kinds of biomasses have been subjected to pyrolysis, including agricultural residues (Aglevor and Besler 1996; Di Blasi et al. 1999), olive husk (Rao and Sharma 1998; Zabaniotou et al. 2000; Demirbaş et al. 2001), and rice husks (Raveendran et al. 1995; Wang et al. 1995; Ahuja et al. 1996). The pyrolysis reaction is a basic step of any combustion or gasification process for utilizing a solid fuel. Solid gasification can be modeled as fuel pyrolysis, which is followed by gasification of the solid residue. Thus, quantification of the product yield from pyrolysis is important for developing a model of the gasification process. In this context, it is useful to estimate the amount of char produced by the pyrolysis reaction.
This study established a mathematical model to predict the rate of carbon derived from the pyrolysis reaction of corn cobs and rice hulls based on the temperature of the reaction at isothermic conditions. The experimental pyrolysis data of corn cobs and rice hulls (treated in a muffle furnace) were used in a polynomial model of the results.

EXPERIMENTAL

The pyrolysis reactions were performed in a muffle furnace. Crucibles with vacuum lids were weighed with and without samples. The oven heating rate was set to reach the given temperature T_c. Once T_c was reached, the crucible with sample was placed inside for 45 min. At the exit of the furnace, the assembly is immersed in an atmosphere of less than 0 ° C. in order to avoid possible reactions inside the crucible. After the sealed crucible cooled completely, its mass was determined. The mass fraction of carbon in the sample at T_c was calculated from the mass before and after pyrolysis. Four replicates were performed at each T_c for both corn cob and rice hull biomasses to determine repeatability. The corn cob and rice hull samples were oven-dried prior to the pyrolysis treatment.

Pyrolysis experiments were conducted at atmospheric pressure at temperatures (T_c) between 350 °C to 600 °C with samples of about 3 g to 10 g. The moisture content of the dried biomasses was determined, and the results reported are based on the dried biomass mass. The experimental scheme is illustrated in Fig. 1.

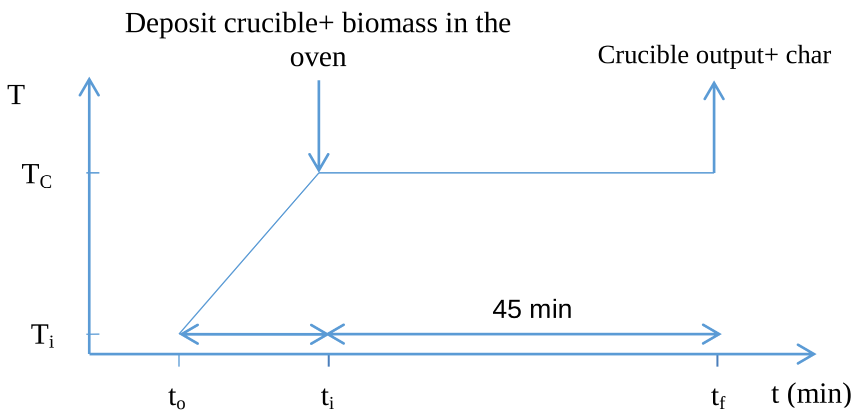

Fig. 1. Schematic of the experimental pyrolysis procedure used in this investigation

Between time t_o and t_i, there is a slow and linear heating rate from starting temperature T_i to set-point temperature T_c. The crucible containing the biomass is maintained at T_c for 45 min. After this isothermal T_c treatment, the crucible with its lid is released and allowed to cool. Then the pyrolysis mass is determined from the sample mass of before and after pyrolysis. The muffle furnace variables are defined as were as follows: T_c, isothermal set-point temperature; T_i, initial sample temperature; t_o, time the oven is turned; t_i, time marking the beginning of the isotherm at T_c of the sample; and t_f, time marking the end of the isotherm treatment of the sample.

Material and Methods

The corn cobs and rice hulls were obtained from Benin, a country located in West Africa. Corn cobs were ground into particles with diameters less than or equal to 4 mm; rice hulls had the rough dimensions of 10 mm x 2 mm. The physico-chemical properties of the studied corn cobs and rice hulls are shown in Table 1. Before each experiment, the tested corn cob and rice hull samples were dried in a ventilated oven at 105 °C to constant mass. The ash content of each biomass was determined in accordance with NF M 03-003 (1968). The volatile matter index was determined by thermogravimetric analysis (TGA) at a heating rate of 5 °C/min under a helium blanket of 12 L/h. Determination of the fixed carbon ratio was performed in accordance with NF M 03-006 (1994). The ultimate analysis was conducted in accordance with NF EN 15104 (2011), while the biomass physico-chemical analysis was performed in accordance to the Van Soest method (Van Soest and Wine 1968).

Table 1. Physico-Chemical Characteristics of Corn Cob and Rice Husk Biomass

Analyses	Components	Corn Cobs	Rice Hulls
		Mass fraction (%)	Mass fraction (%)
Proximate	Moisture content	8	7
	Ash content	1.48	18.31
	Volatile matters	85.05	70.6
	Fixed carbon	13.47	11.9
Ultimate	C	47.5	39.9
	H	5.96	4.77
	O	42.9	35
	N	0.63	0.41
Physico-chemical	Cellulose	35	48.9
	Hemicellulose	31.4	18.4
	Lignin	7.9	14.8

Concerning the chemical composition of the biomasses studied, Table 1 lists mainly the major components (C, H, O and N); The remainder of the percentage is divided between sulfur (S) and chlorine (Cl) which are in inadequate amounts.
The gross errors regarding the experimental data of this study were not included or determined (Bally and Berroir 2008). Systematic errors due to the use of electrical oven muffles, a calibrated scale, and a dry matter with a relative humidity of 9.5% were not examined. However, the random errors due to sudden drop in oven temperature during its opening were noted (Taylor 1996). It is an important parameter that may affect the experimental data.

Statistical Treatment of Experimental Data

A statistical analysis of random errors (Bourdillon 2001) was performed on the experimental data at a confidence interval of 99% to make an objective judgment on the obtained results. Depending on the number of trials (N), two parameters were defined for each temperature: the average ($\bar{X}$) and the standard deviation (σ). The best estimate of the true value X, denoted $\bar{X}$ and obtained from the N measurements X_1, X_2 ... X_n, is the average of these measures (Eq. 1). The best estimate of σ is expressed by Eq. 2.

$$\bar{X} = \frac{\sum_{i=1}^{N} X_i}{N} \quad (1)$$

$$\sigma = \sqrt{\frac{\sum_{i=1}^{N}(X_i - \bar{X})^2}{N-1}} \quad (2)$$

When the sample size is small, the standard deviation is expressed differently by using two parameters: the “t statistic or t student” and confidence limit (CL). This coefficient “t”, tabbed, depends on the number of points averaged and confidence level (Table 2). The confidence limit (p %) used in the treatment of random errors associated with the experimental data is 99%, matching the interval I below in Eq. 3, with the uncertainty defined in Eq. 4.

$$I = \bar{X} \pm \frac{t \times \sigma}{\sqrt{N}} \quad (3)$$

$$\frac{t \times \sigma}{\sqrt{N}} \quad (4)$$

Table 2. P Factor for Confidence Limit (Bourdillon 2001)

Number of measurements N	P Factor for Confidence Limit			
	80%	90%	99%	99.9%
2	3.08	6.31	63.7	637
3	1.89	2.92	9.92	31.6
4	1.64	2.35	5.84	12.9
5	1.53	2.3	4.60	8.60
6	1.48	2.02	4.03	6.86
10	1.38	1.83	3.25	4.78
15	1.34	1.76	2.98	4.14
∞	1.29	1.64	2.58	3.29

Because Table 2 which gives (t) according to the number of trials (N) and confidence limits, the true value of the char mass fraction on an isothermal can be estimated (Table 3).

Table 3. Estimated Value of the char mass fraction of corn cobs from experimental data at 300 °C

	Assay 1	Assay 2	Assay 3	Assay 4	Assay 5	Assay 6
M1 (dry mass entry)	5.00g	5.00g	5.00g	5.00g	5.00g	5.00g
M2 (mass of char)	2.27 g	2.29 g	2.27 g	2.27 g	2.27 g	2.27 g
% (char mass fraction)=$\frac{M_2}{M_1}$	0.454	0.458	0.454	0.454	0.454	0.454
Number of measurements (N)	6					
Average ($\bar{X}$)	0.455					
standard deviation (σ)	0.0017					
Confidence Limit (LC)	99%					
t Student or t Statistic	4.03					
Uncertainty	0.0028					
Interpretation	0.455 ± 0.0028 There is 99% of chance that the true value estimated from the average is within this range: 0.445 ≤ %mchar ≤ 0.458					

Results and discussion

The experimental data is presented in Table 4. The data was curve-fitted to a polynomial function using Newton's algorithm (Fortin 2001) with MATLAB 7.0 software (Breiner and Biran 2004) to minimize the function's lack-of-fit and to estimate equation coefficients.

Table 4. Char Mass Fractions from Corn Cobs and Rice Husk in Accordance to Pyrolysis Temperature

T (°C)	%Char	
	Corn Cobs	Rice Husk
300	0.455	0.643
325	0.404	0.588
350	0.365	0.543
375	0.335	0.519
400	0.314	0.489
425	0.293	0.474
450	0.276	0.469
475	0.263	0.443
500	0.254	0.436
525	0.242	0.414
550	0.236	0.414
575	0.231	0.408
600	0.227	0.399

The function estimates the mass fraction of the biomass char for a given isothermal pyrolysis temperature *T*. In general, the polynomial expression relating *T* to mass fraction of carbon char is given by Eq. 5,

$$\%\ char = a + bT + cT^2 + dT^3 + eT^4 + fT^5 + gT^6 + hT^7 + iT^8 \quad (5)$$

where % *char* is the fraction mass of char (%); *T* is the isothermal pyrolysis temperature (°C); and *a*, *b*, *c*, *d*, *e*, *f*, *g*, *h*, and *i* are coefficients of the fitted polynomial.
The reaction temperature was used to create a function to estimate the char mass fraction resulting from corn cobs and rice hulls pyrolysis. The temperature varied between 300 °C and 600 °C. The fitted curves are shown in Fig. 2. The coefficients of polynomial function for corn cobs between 300 °C and 500 °C and between 500 °C and 600 °C are presented in Tables 5 and 6, respectively. The validation of the mathematical model established was made thanks to the difference observed on the different curves below which show the experimental data and those resulting from the mathematical model. Indeed, we observe a perfect match between experimental data and those from the model. However, there is a small difference between 420 and 500 ° C during pyrolysis of corn cobs; this small difference is due to the large number of points considered in the range [300 °C., 500 °C]. Indeed, the more important the number of points considered, the more error is obtained according to Newton's method (Fortin 2001).

Table 5. Coefficients of the Polynomial Function of Corn Cobs for *T* between 300 °C to 500 °C

i	1.65×10^{-17}
h	-5.329×10^{-14}
g	7.499×10^{-11}
f	-6.004×10^{-8}
e	$29{,}91 \times 10^{-9}$
d	-9494.175×10^{-6}
c	1.875
b	-210.664
a	10,309

Table 6. Coefficients of the Polynomial Function of Corn Cobs for *T* between 500 °C to 600 °C

e	*d*	*c*	*b*	*a*
6.4×10^{-10}	-1.429333×10^{-6}	$1.196799475 \times 10^{-3}$	-0.44549639125	62.468451875

Figure 2 compares the experimental data to the fitted polynomial functions of Eq. 5 for the two different isothermal temperature ranges for corn cobs.

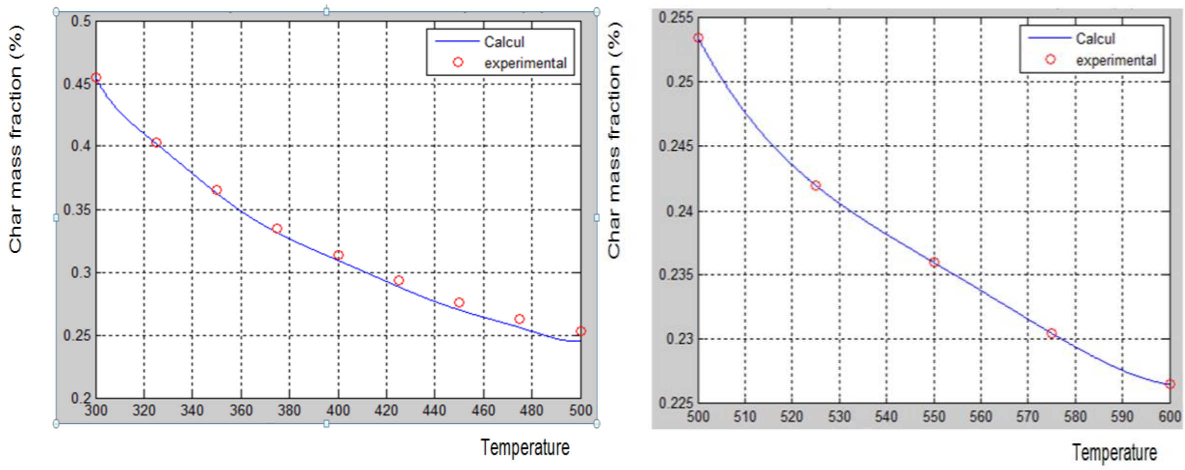

Fig. 2. Experimental data *versus* model predictions of the char mass fraction from the isothermal pyrolysis of corn cobs from 300 °C to 600 °C

The fitted polynomial functions of Eq. 5 for rice hulls at various *T* ranges are presented in Tables 7 to 10. Figure 3 shows the differences noted between the observed experimental data and the fitted curves (of Eq. 5) for rice hulls.

Table 7. Coefficients of the Polynomial Function of Rice Hulls for *T* between 300 °C to 400 °C

e	*d*	*c*	*b*	*a*
-4.544×10^{-9}	6.27813×10^{-6}	-0.00323133675	0.7326836125	-61.04468625

Table 8. Coefficients of the Polynomial Function of Rice Hulls for *T* between 400 °C to 475 °C

d	*c*	*b*	*a*
-3.2587×10^{-7}	0.00042324425	-0.1833831375	26.979155

Table 9. Coefficients of the Polynomial Function of Rice Hulls for *T* between 475 °C to 500 °C

b	*a*
-2.5×10^{-4}	0.56125

Table 10. Coefficients of the Polynomial Function of Rice Hulls for *T* between 475 °C to 500 °C

e	*d*	*c*	*b*	*a*
3.3280×10^{-9}	-7.4488×10^{-6}	0.00624346	-2.322857	324.0998

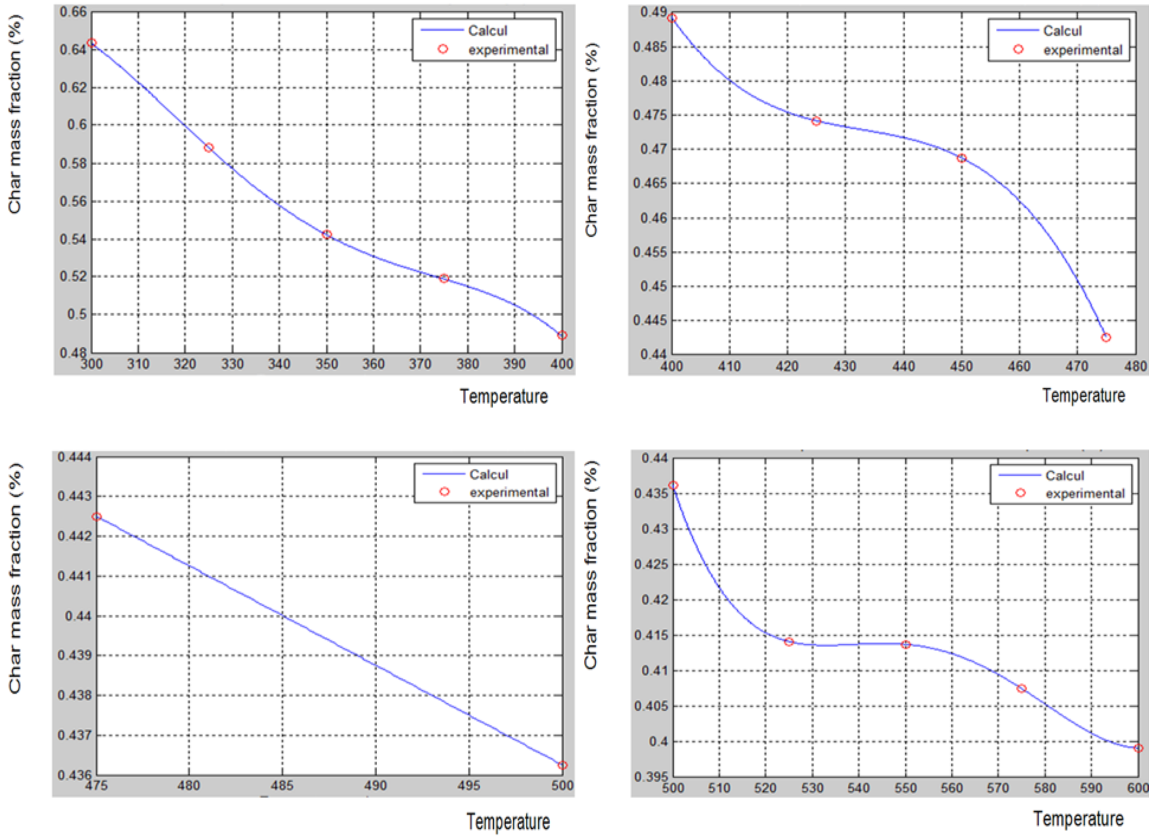

Fig. 3. Experimental data *versus* model predictions of the char mass fraction from the isothermal pyrolysis of rice hulls from 300 °C to 600 °C

Analysis and Comparison

Demiral *et al.* (2012) studied the influence of pyrolysis temperature, the heating rates, and the volume of gas on the pyrolysis products from ground corn cob particles that had diameters between 0.425 mm to 0.600 mm. In dynamic mode, on a fixed bed, for a taken mass of 15 g, the yield of char decreased from 32.16% to 27.19% when the pyrolysis temperature varies from 400 °C to 550 °C at a heating rate of 7 °C/min, while it ranged from 26.89% to 23.29% for a heating rate of 40 °C/min. In the current investigation, the pyrolysis was carried out in a muffle furnace, at isothermal conditions, and for 45 min with corn cob particles with diameters between 1 to 4 mm. The mass fraction of char varied from 31.4% to 23.6% for isothermal temperatures of 400 °C and 550 °C, respectively. Similarly, Demiral *et al.* (2012) identified two thermal decomposition intervals for corn cobs: 350 °C to 500 °C and 500 °C to 600 °C. This was also the case for Cao *et al.* (2004); these authors distinguished two temperature ranges for the thermal decomposition of corn cobs: 350 °C to 400 °C and 400 °C to 600 °C. In Cao *et al.* (2004), the rate of solid residues obtained after 3 to 4 h of pyrolysis was 26% to 32% for temperatures of 600 °C and 350 °C, respectively. However, the char mass fraction was 36.5% at 350 °C in the current study. In studying the effect of temperature on pyrolysis products from corn cob particles, in the order of 1 mm to 2 mm, Zhang *et a*

(2009) obtained 34.2%, 27.0%, 23.2% and 22.0% at 400 °C, 500 °C, 550 °C, and 600 °C, respectively, for a carrier gas flow of 3.4 L/min from a fluidized bed. In our study, these yields are of the order of 31.35%, 25.35%, 23.6%, and 22.65%, respectively without inert gas at the same temperature. Yanik *et al.* (2007) reported a char yield of approximately 23% ± 1.5% at 500 °C for fast pyrolysis in fluidized bed with a 1 to 2 s residence time using agricultural residues ground to a particle diameter of less than 1 mm. In contrast, Demirbaş (2004) observed a 30.6% biochar yield from the 450 °C pyrolysis of ground corn cobs (1.5 to 2.2 mm in diameter); the biochar yield of the current study was 27.6%. Demirbaş (2004) indicated that the char mass fraction decreased with increasing pyrolysis temperature, and ranged from 45.5% (at 300 °C) to 22.65% (at 600 °C). In other words, the pyrolysis conversion rate of corn cobs from the Demirbaş (2004) study increased from 54.5% to 77.35%. According to Zabaniotou *et al.* (2000), this decrease in biochar yield was attributed to a primary decomposition of the biomass or to a secondary thermal decomposition of char. This gradual decrease of the char pyrolysis yield is also accompanied by the production of gas and/or liquid. It's also the case for Demirbaş (2006) who studies the effect of temperature on pyrolysis products of nutshell and shows a decrease in the char yield with temperature increase.

The influence of temperature on the pyrolysis char rate production can be illustrated by the curve of Fig. 3, which illustrates the decrease in the char mass fraction from biomass pyrolysis as a function of the temperature increase. The pyrolysis yield in char was 64.3% at 300 °C, while it's 39.9% at 600 °C for rice hulls. This corresponds to pyrolysis yield of 35.7% to 60.1%, respectively, for these temperatures. Table 11 shows that many researchers are interested in the thermal decomposition of rice husks at different operating conditions and obtained different results.

The char yield results of this study (39.9%) were close to those obtained by Worasuwannarak *et al.* (2007) (39 %), Windeatt *et al.* (2014) (37%), at 600 °C pyrolysis of rice hulls with different particle sizes. However, Hsu *et al.* (2015) indicated that there were three distinguishable temperature ranges depending on the curve obtained for the fast pyrolysis of rice husk using a fluidized bed reactor: 375 °C to 400 °C, 400 °C to 450 °C, and 450 °C to 500 °C. Furthermore, Pattiya *et al.* (2012) described the evolution of char mass fractions for rice hulls pyrolysis obtained from a fluidised bed. In our model, we found however a divergence in the results at interval from 400 °C to 500 °C. And therefore, convergence is achieved when one manages to part the temperature range into two: from 400 °C to 475 °C, and 475 °C to 500 °C. In conclusion, the prediction model of the char mass fraction from the pyrolysis of the rice hulls is established for four temperature ranges: 300 °C to 400 °C; 400 °C to 475 °C; 475 °C to 500 °C; and 500 °C to 600 °C.

The production rate of char from rice hulls is greater than that from corn cobs regardless of the pyrolysis temperature. This observation is attributed to the fact that rice husks contains much higher lignin rate than corn cobs according to the work of Yang *et al.* (2007).

Table 11. Pyrolysis Mass Fractions of Rice Husk Char Reported from Various Literature Studies versus the Current Investigation

Study	Particle Sizes of Rice Husk (Diameter *d* or Dimensions)	Conditions Employed during Pyrolysis	Temperature	Char Mass Fraction	Char Results from the Current Investigation
Worasuwannarak *et al.* (2007)	$d \leq 74$ μm	10 °C/min; 5 ml/min He_2 gas	600 °C	47.0%	39.9%
Windeatt *et al.* (2014)	1.4 mm ≤ d ≤ 2.8 mm	Fixed bed: 5 °C/min heat rate; 200 ml/min N_2; 6 g rice husks	600 °C for 1 h	39.0%	39.9%
Heo, H. S et *al* (2010)	8 to 10 mm long, 2.0 to 2.5 mm width, & 0.1 to 0.15 mm thick	Fluidized bed: 5 L/min N_2; 2.5 g/min to 150 g total rice husks	450 °C	30 to 40%	47.0%
Tsaia *et al.* (2007)	< 0.5mm	Fixed bed: 1 L/min N_2; 200 °C/min heat rate; 1 min residence time; 11 to 21 g rice husks	500 °C	47.1%	43.9%
			600 °C	37.0%	39.9%
Hsu *et al.* (2015)		Fluidized bed: 40 L/min N_2; 10 g/min rice husks feed rate at 600 °C	600 °C	38.5%	39.9%
Williams and Nugranad (2000)	0.25 to 1.0 mm	Fluidized bed: 5 s gas residence time; 0.200 ± 0.015 kg/h rice hulls feed rate with 20 min residence time	400 °C	33.0%	48.9%
			450 °C	32.0%	47.0%
			500 °C	29.0%	43.9%
			550 °C	26.8%	41.8%
			600 °C	25.5%	39.9%

CONCLUSIONS

A mathematical model was developed to calculate the mass fractions of carbon residues (char) from the exothermic pyrolysis of biomass. This formula is based on experimental results obtained from the pyrolysis of corn cobs and rice husks. The developed mathematical formula predicted results close to those obtained experimentally. This mathematical formula can be an important tool to numerically simulate the gasification reaction of corn cob char. The experimental results were in agreement with those reported in the literature, and particularly those obtained from the rice hull biomass. A few differences observed were attributed to the particle sizes of the biomasses, as well as the pyrolysis temperature regime used. So rice husks char is greater than that from corn cobs.

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