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A thermogravimetric model to predict yield product distribution in pyrolysis of agricultural biomass

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ABSTRACT

The present study contributes to the opportunities in producing gaseous or liquid substitution fuels by biomass pyrolysis or gasification. By both processes, the issued fuel characteristics are quite different so that the application fields have to be optimized. Using a predictive model, pyrolysis mass balance could be evaluated so that the fuel qualities could be predicted. In the present study, a predictive model based on proximate analysis of biomass input has been applied on experimental results from three agricultural residues (olive tree cuttings, rapeseed residues and soya residues) which have been pyrolyzed and gasified in a captive sample (fast pyrolysis) and a fixed bed reactor respectively. The experimental results were further compared with the results from the predictive model. The comparison showed deviations ranged between 0% and 28%. The highest deviation appeared in the net calorific value of the char. For the gasification results, on the other hand, the model does not fit the experimental results due to the differences in the assumptions taken. It is only used for the prediction of the heating value of the produced gas.

1. Introduction

The thermal decomposition of biomass which generates degradation products has been largely studied during the last years. Different processes are used and the products issued from different technologies are classified in main three categories gases, liquid and solid. Pyrolysis and gasification are two thermochemical biomass valorisation methods. Pyrolysis occurs in an inert atmosphere at temperatures lower than 800 °C. It can be distinguished as flash, fast and slow depending on the heating rate. Differences between slow and fast pyrolysis are well known [1–3]. On the other hand, gasification takes place at higher temperatures under a flow of O_2/air/mixture, aiming to the optimization and maximization of the gas production [4].

The experimental procedure of both processes can be very complicated and difficult, leading to inevitable human errors. Furthermore, setting up reactors and plants require high finance investments and are time consuming. In addition, there is a strong dependence of the carbonization products in relation with the main process parameters such as final temperature, pressure, heating rate and residence time. The physical and chemical properties of the material are of great importance, influencing the heat transfer from the reactor inside the material.

Therefore, biomass thermochemical conversion development requires methodologies for the prediction of the product distribution in full-scale scenarios. However, this is not easy. Many studies [7–13] have been referred in the literature concerning several predictive models, developed for different purposes and taking different criteria into account for pyrolysis and gasification processes. Considering the complex range of interrelated phenomena, if quantitative accuracy and computational tractability are to be achieved for full-scale applications, some care is necessary in order to ensure an appropriate balance between the level of sophistication in the various component sub-models, and between 'empiricism' and 'fundamentals' [5,6].

As wood is the most common biomass, researchers have done extended predictive studies on its behaviour during thermal treatments. Bilbao et al. [7] studied the ignition of wood, predicting the time to perform it under different operating conditions. Their model was able to predict the temperature profile of the solid and the ignition time of the raw material. Recently, some researchers from China [8] used a modified partial differential equation with the aim to describe a relationship between the charring rate and the increasing rate of heat fluxes and density of wood. Peters and Bruch [9] have also studied wood to develop a flexible and stable numerical method to predict the thermal decomposition of large

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wood particles due to the drying and pyrolysis process. The same team has also developed a model that describes the conversion of wood combustion under fixed bed conditions [10].

Models based on TGA results have also been developed. Such model, semi-empirical, was developed for sawdust pyrolysis [11], where researchers from India, obtained a correlation between the reaction rate constant and temperature. Weng et al. [12] used TGA results of the oxidation kinetics in their model to predict the behaviour of wood pyrolysis. Lee et al. [13] and his team were interested in the gaseous pyrolysis products and focused in the prediction of the gaseous product by employing two computation codes considering thermodynamic and kinetic simulations.

The predictive model applied in the present study is based on the proximate analysis of the biomass input, and it has been developed in order to evaluate the mass balance during pyrolysis and gasification. It is attempting to make easier and less costly the evaluation of the energy and materials production potential from selected agro-residues in Greece. The pyrolysis and gasification experiments were performed in the Laboratory of Chemical Processes and Plant Design in the Chemical Engineering Department of Aristotle University of Thessaloniki (AUTH), Greece, while the predictive model was ran in the Industrial Chemistry Department, Matières et Matériaux, Solvay Brussels School, Centre Emile Bernheim at the Université Libre de Bruxelles (ULB).

2. Methodology and model structure

The model was previously developed and ran at the Université Libre de Bruxelles [17,18] for mixed plastics. The conception of the model is based on the fact that because of the parallel and successive reactions that takes place during the biomass pyrolysis process, the precise mechanism is not clearly known, making the study of the phenomenon extremely hard [14].

The methodology used in the present study was as follows:

1. TGA analysis was performed for two reasons:
   - 1.1 Ultimate and proximate analysis of agro-residues indicates their suitability for energetic valorisation by thermochemical process. The proximate analysis of each fraction of the biomass is needed in order to estimate the mass balances. The elemental analysis (C, H, and O) and each fraction are used to estimate the energy balance.
   - 1.2 Because the operating conditions for the determination of volatile matter are close to the pyrolysis conditions except for the temperature range which is higher (750–800 °C) than pyrolysis (500–650 °C). Therefore, the use of TGA analysis under inert atmosphere is needed to estimate the total devolatilization yield ($\alpha = 1$) and allowing the evaluation of the deviation ($\alpha \leq 1$) of the experimentation. It is well known that the yield of products issued of thermal treatment is dependant on the temperature, residence time and the heat transfer. On the other hand, the devolatilization yield, α, depends also on the heat and mass transfer in the furnace. For rotating kilns, modelling has been assumed at pilot scale unit (CUTEC Institute in Clausthal) [16]. Using the predictive model, the devolatilization yield ($\alpha \leq 1$) will characterise the pyrolysis efficiency.
2. Experiments of fast pyrolysis were performed in laboratory scale, in captive sample pyrolyzer. Gasification experiments were performed a fixed bed gasifier as well as in pilot scale bubbling fluidized bed gasifier. In both cases the produced gas analysed in a GC system and it was concluded that agro-residues produce a medium heating value gas able to be exploited in engines for electricity production.
3. The principle used in the model is based on the ASTM norm for the volatile matter determination [15]. The model estimates mass and energy content of the solid and gases products of the process assuming the additivity of the results of the different fractions of biomass. For gasification, the amount of gas and its heating value was estimated assuming that the char issued from the pyrolysis first stage is oxidized in a second stage into CO, using air based on the amount of carbon present in this specific amount of char.

The model calls upon the proximate analysis of each individual identified fraction of the biomass residue. This model gives the possibility to evaluate a mass balance during the thermal treatment of the biomass residues. Moreover, the ultimate analysis of the fractions of these residues (C, H, and O) enables to estimate the lower calorific value (NCV) of the char (semicoke emerging from pyrolysis) and produced gases resulting from pyrolysis and gasification. The heating values of the produced gas were estimated by Eq. (1) taken from the literature [21], was based on the %vv concentration of gas analysis. The model is using the hypothesis of additivity of the behaviour of each component during thermal decomposition, assuming that each fraction behaves independently. Its component is characterised by its proximate analysis: dry matter (DM), volatile matter (VM) and ash (A) on dry basis.

Using the predictive model, the devolatilization yield ($\alpha \leq 1$) will characterise the pyrolysis efficiency. Taking into account the proximate analysis of the input material (water content W, volatile matter VM and ashes content A), it is possible to estimate the fixed carbon (C_F^*):

$$C_F = 100 - VM - A \quad (1)$$

and the dry matter

$$(DM = 100 - W). \quad (2)$$

Assuming that, during slow pyrolysis, the volatile matter is oriented with the water in the gas phase and that the fixed carbon is recuperated in the solid phase with the ashes, the mass balance could be estimated:

Mass of gas phase:

$$M_G = \alpha VM + W \quad (3)$$

with

$$VM = VM^* \frac{DM}{100} \quad (4)$$

Mass of solid phase (char):

$$M_S = C_F + A + (1 - \alpha)VM \quad (5)$$

with

$$A = A^* \frac{DM}{100} \quad (6)$$

Table 1
Ultimate analysis and proximate analysis of agricultural residues.

Raw material	Olive tree prunnings	Rapeseed residues	Soya residues
Ultimate analysis			
Carbon, C [wt.%]	47.27	44.52	43.59
Hydrogen, H [wt.%]	6.41	5.53	5.6
Oxygen, O[a] [wt.%]	46.32	49.95	50.81
Proximate analysis			
Moisture [wt.%]	4.79	2.2	2.5
Volatiles [wt.%]	74.42	71.4	70
Fixed carbon [wt.%]	20.17	23.42	24.06
Ash, A % [wt.%]	0.62	2.98	3.44
Dry matter, DM [%]	95	98	97

[a]Calculated by difference.

Table 2
Validation of the model for fast pyrolysis experiments of olive tree pruning, rapeseed residues and soya residues.

$\alpha = 1$	Olive tree pruning			Rapeseed residues			Soya residues		
	Experiments	Model	Dev.%	Experiments	Model	Dev.%	Experiments	Model	Dev.%
Kg char/$t_{biomass}$	250	244	6	280	280	–	267	293	10
%ash/char	2.7	2.4	10	10.6	10	2	13	11.5	11
HV char (MJ/kg)	25	32	28	24	29	24	23	29	25
Kg Gas/$t_{biomass}$	740	756	2	680	720	6	724	708	2
% VM/gas	93.6	94	0.1	91.6	97	6	90	96	7
HV gas (MJ/kg)	18.6	18	4	17.5	16	9	19	15	19

It is assumed that the carbon content (C_{VM}) and the hydrogen content (H_{VM}) of the volatile matter (VM) are the following:

$$C_{VM} = C_{DM} - C_F \quad (7)$$

$$H_{VM} = H_{DM} \quad (8)$$

The net calorific value (NCV) of the volatile matter is estimated in the pyrolytic gases based on the use of combustion heat values of light hydrocarbons as:

$$NCV_{VM} = f(C_{VM}, H_{VM}, O_{VM}) \quad (9)$$

The hypothesis for the evaluation of the char requires the experimental value of α obtained by TGA measurements. The volatile matter remaining in the char is then:

$$VM_{char} = (1 - \alpha)VM \quad (10)$$

Subsequently, the fixed carbon in the char will be evaluated:

$$C_F + VM_{char} + A \quad (11)$$

Finally, the net calorific value of the char is estimated by the sum of ponderated values of the NCV of the carbon and the volatile matter in the char:

$$NCV_{char} = \%C_{char}NCV_C + \%VM_{char}NCV_{VM} \quad (12)$$

2.1. Application of the model to fast biomass pyrolysis

The model has been used previously for slow pyrolysis. In this study an attempt is making to evaluate whether the model could agreed with fast pyrolysis experimental results.

During the pyrolysis, partial devolatilization of the total organic matter (MO) of biomass is taking place. The mass balance is estimated, assuming that during slow carbonization at low temperature, the volatile matter is oriented with the water in the gas phase and that the fixed carbon is recuperated in the solid phase with the ashes. The kinetics of the reaction is globally represented in the devolatilization yield (α). Indeed, if the temperature and/or the residence time are too low, some of the volatile matter will remain in the char. If φ is the proportion of the volatiles VM trapped in the char, the output of the devolatilization process will be: $\alpha = 1 - \varphi$.

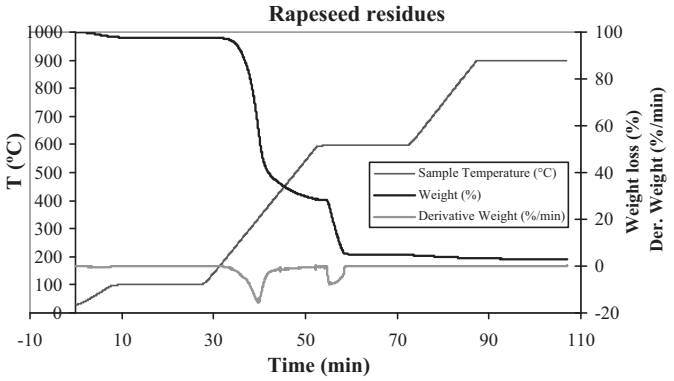


Fig. 1. TGA diagram of rapeseed residues.

The carbonization yield could be found by TGA analysis in low heating rate for the same input material. The calorific value of the char is calculating according to its carbon content without volatile matter (fixed carbon). In order to evaluate the energy balance, the NCV of the pyrolysis gases can be determined using elemental analysis (C, H and O) of the dry matter. The thermodynamic data, such as enthalpies of organic components containing C, H and O and the heat of combustion, are used in order to estimate the NCV of the pyrolysis gases. For a carbonization yield less than 10%, the NCV of the char is evaluated as the sum of the NCV value of the fixed carbon and the NCV of the entrapped volatile matter. An estimation of the net calorific value (NCV) of the pyrolysis gases can be evaluated based on the data of the ultimate analysis (C, H, and O) of the dry matter.

2.2. Application of the model to biomass gasification

In the gasification processes, pyrolysis is followed by a partial oxidation of the fixed carbon content of the char into carbon monoxide in one or two stages, and other permanent gases of biomass, by a strictly controlled air input. The resulting CO mixed with nitrogen ballast, flows currently towards the charge input and is mixed with the pyrolytic gases generated in the first section (the pyrolysis step), while inert ashes are released.

By simulating the one stage gasification process under the current predictive model, the air input was calculated taking into account the carbon content of the char. The nitrogen ballast and the quantity of CO were determined. The composition of the gases at the furnace output was also determined (pyrolytic gases, CO and nitrogen) in order to calculate their NCV.

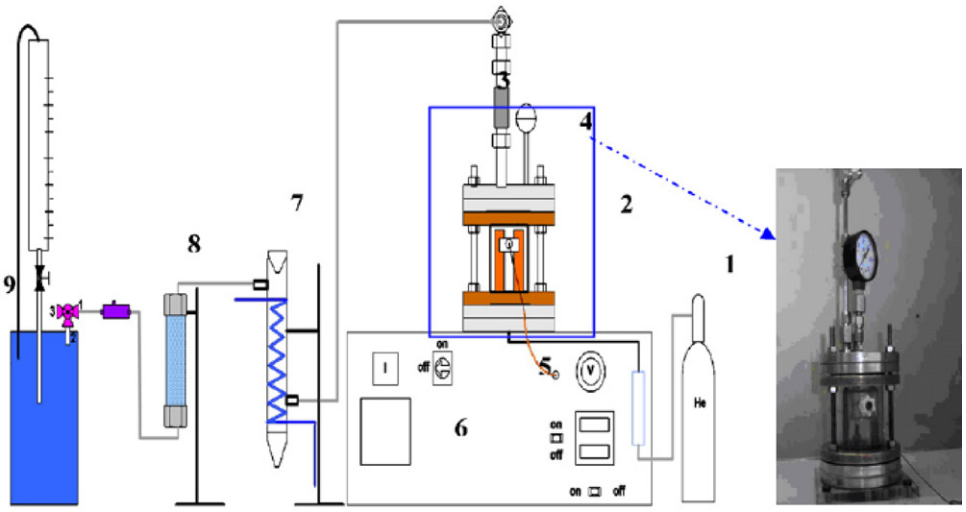
3. Experimental

The raw materials used for experimentation were three various agricultural residues (olive tree prunnings, rapeseed and soya residues) from Greece. Prior to use, samples were washed from dust, grounded to desirable particle size (by a small laboratory scale mill) and sieved to powder of sizes <1 mm in diameter for enabling running the tests. A Thermofinnigan, CHNS, EA 1112 elemental analyzer was used to estimate their ultimate analysis (Table 1).

The proximate analysis was performed by thermogravimetric analysis (TGA) while monitoring the weight change that occurred

Table 3
Composition of the pyrolytic gas.

%v/v Norm (on pure gas)	Olive tree pruning	Rapeseed residues	Soya residues
H_2	35	28	41
CH_4	13	13	10
CO	47	43	43
CO_2	5	16	6



1. He bottle
2. Captive sample reactor
3. Filters
4. Manometer
5. Thermocouple
6. Electrical circuit
7. Water cooling coil
8. Moisture traps
9. Gas collection system

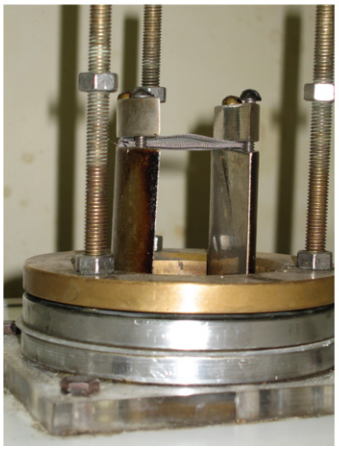


Fig. 2. The experimental set up of the captive sample reactor.

during the sample heating and the fraction of volatile components was determined (Table 1). The instrument used in this study was a Perkin Elmer thermogravimetric analyzer TGA 6, in which the sample was heated under inert atmosphere according to the following temperature profile: heating started at 30 °C with a slow rate of 10 °C min^{-1} and stabilized at 100 °C for 20 min. Then, with a rate of 20 °C min^{-1}, the temperature increased up to 600 °C. According to DiBlasi [6] char formation is an exothermic reaction, whereas tar formation is endothermic. Generally, from thermogravimetric analysis it was observed that the exothermic reaction of char formation stopped at 600 °C and above this temperature there was only a weak endothermic peak representing the tar formation. Thus, it was assumed that when the temperature is 600 °C there is no volatile matter in the char ($\alpha = 1$). Therefore, during the TGA in the current study, air was introduced at 600 °C and oxidation was occurred up to 900 °C with a rate of 20 °C min^{-1}.The first weight loss phase was due to the water layer evaporation from minerals. For temperatures up to 400 °C lighter volatiles identified as hemicellulose and cellulose were decomposed. Heavier volatiles, such as lignin decompose, were identified up to 600 °C. For temperatures higher than 600 °C, where oxidation occurred, the fixed carbon was determined. Thus, the amount of mass at the time the decomposition of hemicelluloses started minus the amount of mass at the time char oxidation started constitutes the total amount of volatiles. The remaining solid at the end of the oxidation (900 °C) phase was ash. A TGA diagram of rapeseed residues is shown in Fig. 1.

This experimental work was performed in the Laboratory of Chemical Process and Plant Design in the Chemical Engineering Department of Aristotle University of Thessaloniki.

3.1. Pyrolysis in a captive sample reactor

The experimental apparatus included a wire mesh sample reactor comprised two electrodes which are made of a nickel and bronze alloy, an electrical circuit, water cooling coil, a trap for moisture, two filters for liquid hydrocarbons, a system for gas collection and a system of gas analyses (GC). The sample that placed inside the wire mesh was weighted ~0.3 g. The particle size of the samples was 1 mm. A thermocouple was placed inside the sample and was connected with a computer where through a program it gave the ratio between temperature and time (heating rate). In the current study the pyrolysis experiments occurred at 600 °C, as it was assumed

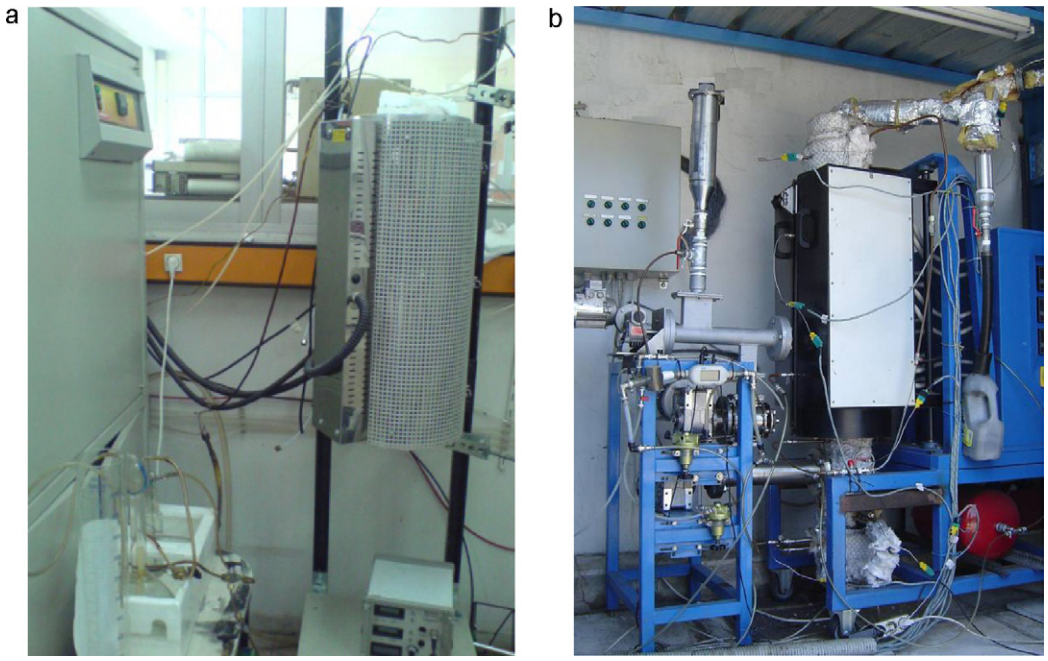


Fig. 3. (a) Experimental set up for laboratory scale gasification of agro-residues. (b) Experimental unit for pilot scale gasification of agro-residues.

that $a = 1$ at that temperature. The inert atmosphere was achieved with He (flow of 30 ml/min), at atmospheric pressure.

The pyrolysis products were, char, gas and liquids. The solid product (char) remained in the screen and was determined gravimetrically. The liquid product was comprised by tar and liquid hydrocarbons condensed within the reactor vessel, on the wall and flanges of the reactor and on the paper filter at the exit of the reactor and measured gravimetrically. The gas was collected in the water container. The volume of the water that was displaced determined the gas volume. Each experiment was repeated three times. The reactor and the whole procedure are presented detailed elsewhere [19]. A schematic representation, though, is given in Fig. 2.

3.2. Gasification in a fixed bed reactor

The gasification experiments were performed with air in a laboratory scale, batch and fixed bed reactor. The experimental set up is presented in another study [20]. The experimental temperature was 750 °C and the amount of biomass placed in the reactor was 1 g. The particle size of the samples was 1 mm. Air, at ambient conditions, was introduced into the reactor moving downwards to the fixed biomass particles. The air factor λ, the ratio of air used in comparison to the air that would be necessary for complete combustion, in the gasification experiments was kept at $\lambda = 0.42$. The gasification apparatus used are shown in Fig. 3.

4. Model validation

The data that were needed in order to run the model are shown in Table 1. Ultimate analysis (C, H, and O) of the raw materials was used to estimate the energy balance (as described above in the methodology of the model). Proximate analysis was used to recalculate the results on dry basis. The comparison of the results for the three agricultural residues between experimental values and these predicted by the model for pyrolysis experiments are presented in Table 2. The concentration of the gas analysis is summarised in Table 3.

For pyrolysis, the temperature in the experiments was kept at 600 °C which is sufficient to have zero amount of volatile matter in the char for TGA as well, as mentioned above. From the comparison of the pyrolysis results with the literature data and other studies performed by the authors [19,22–25] it is observed that the yields fluctuate in the same area. According to the literature the increase of temperature and heating rate results in the decreasing of the char yield, as further decomposition occurs [24], and the char contributes to secondary cracking in the vapour phase [26]. In the present study though, the amount of the pyrolytic char, given by the captive sample reactor, appeared slightly high. The specific reactor used in these experiments have shown in the past [27], that the char decreases more above 600 °C, and the estimated yields were almost the expected ones. However, when fast pyrolysis is the process studied, there is always a chance that the char might be slightly higher, as it also includes high-molecular-weight tar components [28]. This can be due to the immediate decrease of the temperature, which can cause tar condensation on the char.

An acceptable agreement was obtained through the comparison between the predictive model and the pyrolysis results for the three samples. The deviations ranged between 0% and 28%. The higher deviations were observed in the heating values of the char. The amount of carbon in the char, resulted from the experiments, was found slightly lesser than the expected FC ($\alpha > 1$), which might explain a possible activity of secondary reactions not taken account in the mathematical description.

For the gasification, the experimental results are presented in Table 4. It can be seen that an important quantity of solid remained after gasification of these three residues which means that they were not completely oxidized. Hypostoichiometric amount of air needed was used to avoid the complete oxidation of biomass. The air factor in these experiments was kept steady at $\lambda = 0.42$. These

Table 4
Gasification results for olive tree pruning. rapeseed residues and soya residues ($\lambda = 0.42$, $T = 750$ °C).

	Olive tree pruning	Rapeseed residues	Soya residues
Kg char/$t_{biomass}$	347	284	330
Kg gas/$t_{biomass}$	1114	1183	1157
HV gas (MJ/kg))	7	8	7

Table 5
Estimated gasification values using the predictive model for olive tree pruning, rapeseed residues and soya residues.

	Olive tree pruning	Rapeseed residues	Soya residues
Kg ash/$t_{biomass}$	6	30	34
Kg gas/$t_{biomass}$	2136	2342	2367
HV gas (MJ/kg))	21.6	21	21

conditions do not correspond to the hypotheses made to run the model. Table 5 presents the calculated values using the predictive model. These values cannot be compared with the experimental values shown in Table 4. In fact, the model assumes a two step process: complete oxidation of the charge followed by air gasification. Basic assumption was the use of the exact stoichiometric air amount for partial oxidation of the fixed carbon of the char into CO. So, the predicted model is of interest to indicate mass and energy balances in the best conditions. It shows the optimum potential quantities and energetic values of the syngas that can be produced by air gasification (~20 MJ/kg). The experimental results show about the same quantities of syngas with lower calorific values (~7 MJ/kg) due to the higher value of the air factor and nitrogen dilution of syngas.

Concerning the experimental procedure, the efficiency of the global process depends not only on the material transport into the reactor, but also on the heat transfer to and inside the material. The feed preparation is then essential as well as the characterisation of the eventual side material.

5. Conclusion

For the specific energy needs of Mediterrannean and developing countries a versatile gas-producing system from biomass in conjunction with an engine sounds ideal in terms of ease operation and high efficiency. However, before going ahead with the direct application of such systems it is useful to estimate the product yields using a simple predictive model. The development of such a model has been attempted in this study.

The comparison of the results generated by the model, with the pure experimental results emerged from pyrolysis and gasification processes of the three selected biomass samples olive tree prunnings, rapeseed residues and soya residues showed, that despite the fact that the model describes better slow pyrolysis, it could also applied to the experimental results taken from fast pyrolysis but with bigger deviations. More specific, rapeseed fast pyrolysis experiments showed better agreement with the model than olive tree pruning or soya residues did.

The present study showed that the model described and developed in Universite Libre de Bruxelles could be used for fast pyrolysis but it cannot describe gasification adequately. In other to use the model for gasification, further experimental results are needed from a two stage process comprising pyrolysis and char oxidation instead of one stage process studied here.

The application of the model for mixtures of plastic waste has already previously been proved for slow pyrolysis. However, further experiments are planned to confirm the validity of the model for mixtures of agricultural residues.

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