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Pyrolysis products from different biomasses: application to the thermal cracking of tar

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Abstract

The purpose of this study was to evaluate the amounts of various pyrolysis products (gases, water, tar and charcoal) from three biomasses (wood, coconut shell and straw) and to suggest a kinetic equation for the thermal cracking of tar at temperatures varying from 400 to 900°C. From the results, a comparative analysis is done for the biomasses, and a kinetic model of thermal cracking of tar is proposed for a residence time ranging from zero to 4s . This can be applied to the purification of gasification gases used as a feed gas to a combustion engine, and so contributes to the design of gasifiers. © 2001 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Pyrolysis of ligno-cellulosic biomass is a very complex process of interdependent reactions; nevertheless it can be reduced to the reaction illustrated in Fig. 1, universally known as the Broido–Shafizadeh mechanism [1,2].

Secondary reactions are related to the thermal degradation of volatile tars. Strong interactions occur during secondary reactions. Thus, it was well established, particularly by Antal [3], that more solid char is formed if the volatile compounds constituting tar are confined within the solid matrix, by increasing the pressure or by slowing down the heating rate. The distinction is commonly made between flash (or fast) pyrolysis and slow pyrolysis. Flash pyrolysis is performed on a finely-ground material at a high heating rate (500 to 1000°C/min) and the yield in tar products can be as high as 60–70% without char formation. Slow pyrolysis is performed on a coarse material or at low heating-rates yielding a solid char of up to 35%. Concerning the thermal degradation of biomaterials, most of the investigations focused

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on their major component : the cellulose. The decomposition of cellulose at low temperature leads to two groups of basic compounds:-

- monomeric volatile sugars, such as levoglucosan, resulting from a total depolymerisation of cellulose
- partially-depolymerized compounds, called anhydrocellulose, which are precursors of the residual solid char.

The subsequent degradation of the primary basic compounds at higher temperatures leads to products distributed among various fractions or physical phases: solid char, light gas, water fraction and tar fraction. Actually, only a few studies have been devoted to the quantitative analyses of these fractions. Some of them were conducted at a laboratory scale. Déglise et al. [4] pyrolyzed fine beechwood and pinewood particles, in a small tubular oven heated to between 500 and 1000°C. Beaumont and Schwob [5] used approximately the same technique to pyrolyze wood granules up to only 500°C and Henriksen et al. [6] to pyrolyze barley straw at 600 and 900°C. With a larger experimental set-up, Shah et al. [7] studied the slow pyrolysis of wood blocks in a 10 l retort, up to 650°C, in order to assess the quality (fixed carbon, friability, etc.) of the produced charcoal. Kosstrin [8] described the variations of the decomposition fractions with temperature in the case of the ERCO's pyrolysis facility, using a large capacity fluidized-bed to process various cellulosic wastes. In order to determine the conditions which maximize tar production, Scott, Piskorz and Radlein [9,10] achieved flash pyrolysis of various cellulosic biomasses, in a mini-fluidized bed of sand fed with up to 3 kg/h of dry biomass, at moderate temperatures ranging from 400 to 600°C.

A knowledge of the kinetics of pyrolysis reactions is useful for the monitoring of commercial pyrolysers or gasifiers. For pure cellulose, the kinetics of the primary reactions, as defined in Fig. 1, have been extensively studied. By comparison, the secondary reactions of tar decomposition are less well understood; the most pertinent investigations being due to Kosstrin [8], Diebold [11], Liden et al. [12], Boroson et al. [13]. These authors published exploitable values of the pre-exponential factor and activation energy of the reaction of tar decomposition.

For practical and environmental reasons, emissions of volatile tar are undesirable and many inorganic compounds have been tested with the aim of reducing tar

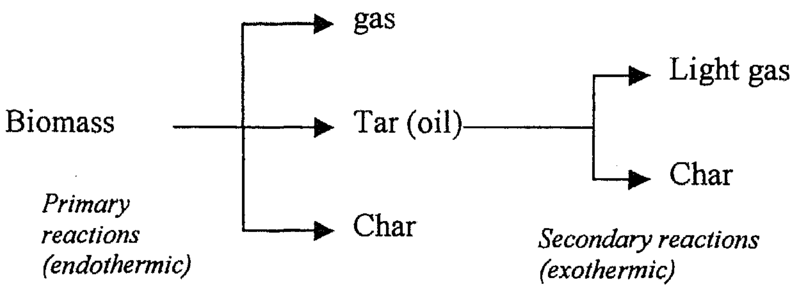

Fig. 1. Overall reaction mechanism of pyrolysis.

concentration by catalytic cracking. Calcinated dolomite has the most efficient effect and the tar content after treatment with dolomite is often reported to drop almost to zero [14].

In the present work, the cracking of emitted tar is considered as a thermal process, without catalytic effects, as far as the emitted gas is put in contact with a packing of metallic rings which induce a strong turbulence in the flowing of gas without too great a pressure drop as in a bed of dolomite.

2. Materials and methods

2.1. Experimental device

The test rig for pyrolysis experiments is illustrated in Fig. 2. The reactor is made of a quartz tube, 1.20 m in length and 6 cm in diameter, heated by a cylindrical oven, the temperature of which is regulated. The bottom of the reactor is packed with Pall rings of stainless steel, having a variable depth from zero to 30 cm. The volatile compounds generated in the oven are aspired by the circulation pump at the bottom of the reactor. Mixed with the carrier gas (helium), they successively flow through two cooled condensers, a filter, a $CaCl_2$ dryer, before being re-injected in the reactor. During pyrolysis, the condensable volatile products are trapped in the cooled condensers while the non condensable light-gas fraction is collected in a swelling rubber bag, which is empty at the start of the experiment. The total volume of the light-gas fraction entering the rubber bag is measured with a gas meter.

2.2. Experimental procedure

This can be described by the following successive operations:

- a metallic mesh basket containing 20 to 30 g of ligno-cellulosic material is first placed in the unheated upper part of the reactor;
- the oven is switched on and its temperature regulated, at a fixed value between 500 and 1000°C;
- the test rig is purged of air and filled at atmospheric pressure with helium, the carrier gas. The circulation pump is then switched on; and
- the basket is let down into the heated part of the reactor, so that the pyrolysis reaction starts.

At the end of the pyrolysis, the basket is lifted again into the upper part of the reactor. A sample of the light-gas fraction is taken and analysed by a gas chromatograph equipped with a catharometric detector and two separation columns, one packed with a molecular sieve, the other with porapak "S"; the carrier gas in the chromatographic columns being the same as in the test rig, i.e. helium.

The total condensed fraction, consisting of tar and the water fraction, is first weighed and then recovered by dilution in acetone. The vaporisation of the solvent at 70°C under reduced pressure yields a viscous residue: the tar fraction. The carbonaceous solid residue remaining in the basket is recovered and weighed.

During pyrolysis, the residence time of the volatile products in the cracking zone (packing) can be fixed to be between a minimum value of about 0.3s to a few seconds by varying the depth of the packing and the flow rate of the gas-circulation pump. The cracking time is defined by the ratio:depth of cracking zone/velocity of circulating gas.

Prior to pyrolysis, the raw biomass is not milled into the form of fine particles i.e. sawdust or powder, as for the fast pyrolysis processes. Wood is simply cut into chips of 3 to 5 mm in thickness. Coconut shells are broken into small fragments with a hammer, and straw wisps cut into fragments of about 10 cm length.

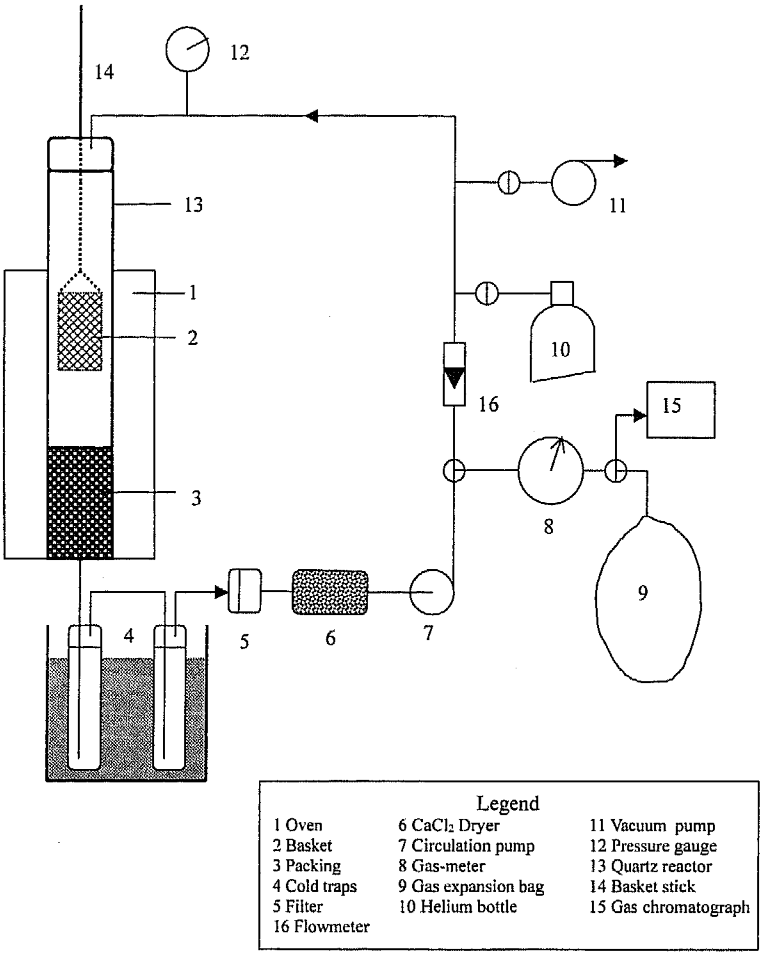

Fig. 2. Schematic diagram of the test rig.

The moisture content of the raw biomass is determined after 3h drying in an oven at 105°C. All the ultimate elemental analyses are performed at the Laboratoire Central de Microanalyse du CNRS (Solaize, France).

The high heating value (HHV) of the raw biomass and pyrolysis products are calculated by means of the following IGT (Institute of Gas Technology) formula, in which the amounts of the elements (C, H, O, N and ash) are expressed in mass percentages:-

$$\text{HHV (kJ/kg)} = 354.68\,\text{C} + 1376.29\,\text{H} + 71.26\text{-}15.92\,\text{Ash}\text{-}124.69(0 + \text{N})$$

3. Results and discussion

3.1. Gas production

The total volume of gas produced is illustrated in Fig. 3. Above 500°C, a sharp increase is observed for the gas volume, up to about 0.48–0.55 Nm^3/kg dry biomass at 900°C, which corresponds to a mass yield of 45–50% with respect to the raw dry biomass. In the case of flash pyrolysis, the gas fractions measured by other investigators are often larger. Thus, for the yield of gaseous products at 900°C, Déglise et al. [4] report values of about 60% and Kosstrin [8], values in the range 60–75%. Gulyurtlu et al. [15] obtained gas yields slightly less than 40% by pyrolysing, at 900°C, wood slabs (pine and helm oak), 0.25 cm in thickness. Straw appears to yield more gas than the other tested bio-materials, namely wood and coconut shells. The low thickness of the wall of the straw wisp, compared with the others bio-materials, results in a higher heat-transfer, and hence a higher rate of pyrolysis, which is favourable to gas and tar production. For the pyrolysis of barley straw at 900°C, Henriksen et al. [6] found 0.48 Nm^3/kg dry biomass, a value very close to that proposed in the present work.

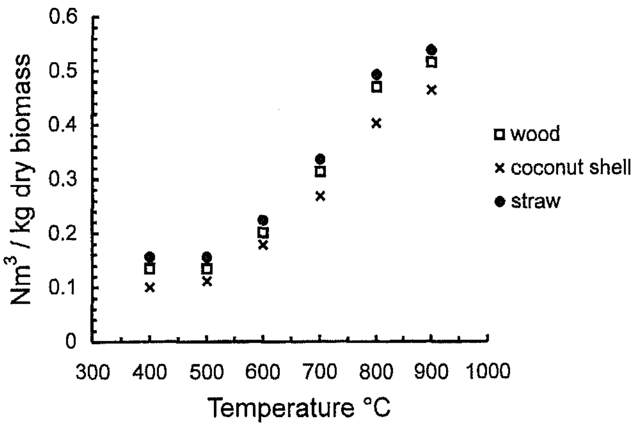

Fig. 3. Gas production as a function of temperature.

Unlike the gas volume which regularly increases with temperature, the heating value of the gas fraction stabilises above 700°C, at a value around 15–16 MJ/Nm^3. Déglise et al. [4] found a heating value reaching a maximum of 20 MJ/Nm^3 at 750°C, then decreasing to 17–18 MJ/Nm^3 at 900°C.

Apparently, the composition of the gas fraction versus temperature between 500 and 900°C does not vary a lot with the kind of tested bio-material (wood, straw and coconut shell), as indicated on Tables 1 and 2. The concentrations of CH_4 and C_2H_x reach a maximum value at about 750°C, that is in good agreement with the results of other investigators [4,16], who found values of CH_4 content of about 13–15% between 700 and 800°C. A regular decrease in CO_2 concentration with temperature occurs with a simultaneous increase in CO and H_2 concentration. High temperatures are known to favour the production of H_2 to the detriment of higher hydrocarbons (in C2, C3) which are dehydrogenated by thermal cracking. The evolution of CO and CO_2 concentrations are consistent particularly when considering the heterogeneous gas-solid reaction at thermodynamic equilibrium, i.e. $C + CO_2 = 2\ CO$; an increase in temperature results in a larger concentration in carbon monoxide.

3.2. Char production

The yield in solid residue regularly decreases with increasing temperature. At 900°C, it is between 21 and 30%, depending on the type of biomass. At a given temperature of pyrolysis, the yield of residual solid appears as:

Straw > coconut shell > wood.

Table 1
Yields of the major gases (in % volume; A, wood; B, coconut shell; C, straw)

		T (°C)					
		400	500	600	700	800	900
CO	A	34.2	39.7	42.5	44.3	50.2	53.5
	B	31	35.0	38.1	40.1	44.2	–
	C	–	35.0	37.7	41.0	48.1	53.3
CO_2	A	51.9	36.6	23.0	16.7	9.1	5.0
	B	53.1	42.2	28.7	17.9	9.8	–
	C	–	40.7	30.0	15.8	8.4	4.5
H_2	A	1.3	7.6	10.8	15.5	20.8	25.3
	B	1.0	5.4	12.4	18.5	23.5	–
	C	–	7.4	12.8	19.2	23.4	24.6
CH_4	A	9.3	12.8	16.5	16.1	14.2	12.1
	B	10.2	13.2	16.8	17.2	15.8	–
	C	–	11.8	13.0	15.3	13.7	12.1
C_2H_x	A	3.3	3.3	7.2	7.4	5.8	4.1
	B	4.7	4.2	4	6.3	5.3	–
	C	–	5.1	5.5	8.7	6.5	5.5

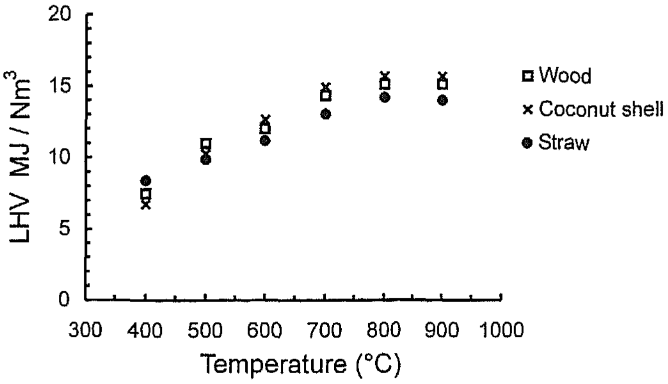

Fig. 4. Low heating value (LHV) of gas as a function of temperature.

The relatively high yield for straw can be explained by the high ash content of this material (see Table 2).

3.3. Tar production

The influence of temperature on tar production is shown in Figs. 4–7 for the three types of biomass with minimal residence times of the volatile products in the reactor. The evolution of tar production is similar for the 3 tested biomasses. The quantity of tar reaches a maximum value at about 500°C, and then drops with increasing temperature. At temperatures higher than 600°C, the secondary reaction (i.e. tar cracking) prevails, leading to a larger amount of gas. Moreover, the cracking of tar improves the energetic content of the product gas. The heating value and therefore the elementary composition of the recovered tar depends on the nature of the raw

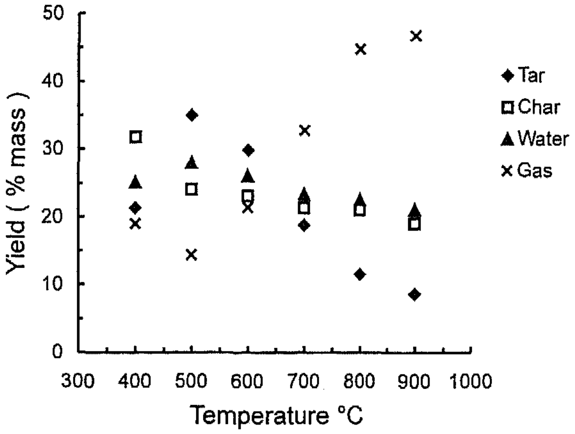

Fig. 5. Wood pyrolysis: effect of temperature on the decomposition products.

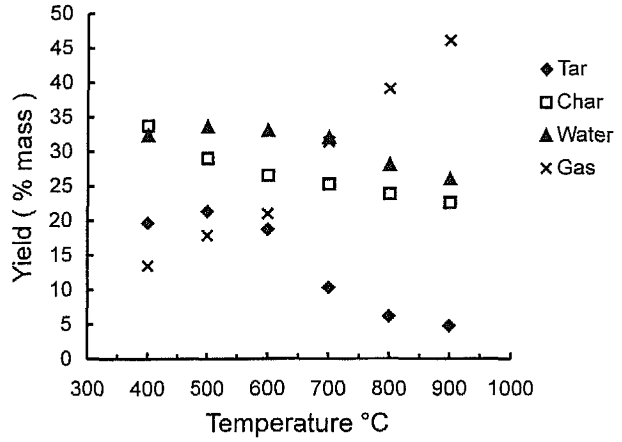

Fig. 6. Coconut shell pyrolysis: effect of temperature on the decomposition products.

biomass. As shown in Table 3, the theoretical and experimental heating values are very close.

The tar yields are in agreement with the values found by Kosstrin who made an extensive experimental study of the fluidized-bed pyrolysis of wood sawdust to generate energy cleanly. He concluded that secondary reactions play a major role in the production of pyrolytic oil. He tested various materials, such as paper, sawdust and corncobs. For all the tested materials, he clearly established that the oil yields decrease with increasing temperature above 500°C. The same behaviour was observed by Scott, Piskorz and Radlein [9,10], who have built and used a fluidized-bed reactor to produce pyrolytic oils from forest residues. By varying the temperature from 400°C to 650°C, they found the maximum tar production occurred at about 500°C.

Table 2
Elemental compositions of solid fractions

Sample		C	H	O	N	Ash	HHV kJ/kg
Wood	Virgin	45.68	6.3	47.42	0.3	0.3	18,988
	500°C	88.51	2.78	8.12	–	0.59	34,268
	700°C	93.22	1.09	4.6	–	1.09	34,043
	900°C	93.18	0.62	5.1	–	1.10	33,320
Coconut shell	Virgin	47.97	5.88	45.57	0.3	0.5	19,450
	500°C	87.09	2.6	8.88	–	1.43	33,408
	700°C	91.83	1.05	5.71	–	1.41	33,352
	900°C	92.02	0.7	5.55	–	1.73	32,953
Straw	Virgin	42.69	6.04	47.11	0.46	3.7	17,534
	500°C	69.38	2.23	9.82	–	16.25	27,088
	700°C	71.7	1.07	13.12	–	16.43	24,254
	900°C	71.63	1.15	9.86	–	17.36	25,553

Table 3
Elemental composition of tar obtained at 500°C

	HHV (kJ/kg)				
	C	H	O	Measured	Calculated
Wood	53.9	6.8	39.3	22,196	23,647
Coconut shell	57.3	6.3	36.4	24,223	25,214

The maximum yield of tar product seems to depend greatly on the type of biomass. The higher yield is 35% for wood, and around 22% for the other materials at 500°C without subsequent thermal cracking of the tar. According to Kosstrin, the tar yield is closely related to the alphacellulose content of the material. Thus, he found a yield of 60% for paper and only 30% for sawdust. The same phenomenon was observed by Sadakata et al. [17], who investigated the conversion of wood, lignin and holocellulose into gas and solid fuels at a heating rate exceeding 1000°C/min. The maximum tar yield obtained by these authors was larger for holocellulose than for other materials. From Samolada and Vasalos [18], an interesting observation reported in the literature and regarding the effect of chemical composition is that samples with the same ash and alpha-cellulose contents give identical yields of pyrolysis liquids. Samolada and Vasalos obtained the maximum yield of pyrolytic oil (56%) at 500°C, as in the present work, by pyrolysing, in a fluidised bed, the fine particles (0.36–0.8 mm) of fir and poplar wood.

3.4. Thermal cracking of tar: kinetic analysis

Tar cracking produces a purified gas, which can be used as feedstock for combustion engines. All the experiments clearly show that the thermal cracking increases the yield of gas, but has actually no effect on the composition of the gas except at higher temperature ($>700°C$), where an increase in hydrogen is observed (see Table 1). The thermal cracking of tar is effective above 500°C. At these temperatures, the yield of tar reaches a maximum value. Modelling the tar cracking phenomenon is of interest above 500°C (the operating temperature of an industrial gasifier). In the experiments of tar cracking, two parameters have been varied: the temperature of the metallic packing (from 400 to 900°C) and the time of cracking (from 0.3 to 4 s). The model is based on two relationships:

- The first expresses the maximum yield of tar instantaneously generated at a given temperature, i.e. for a cracking time equal to zero.
- The second gives the conversion of tar as a function of the residence time of gas throughout the packing.

At a given temperature T, the maximum yield of tar is obtained for a contact time zero between the escaping volatile and the packing. This yield, termed $X_g(T, 0)$ is evaluated from the following relationship:-

$$X_g(T,0) = X_g(500)\exp(-A(T-500)) \tag{1}$$

where T is the temperature in °C and $X_g(500)$ represents the maximum yield of tar produced at 500°C. This yield, as well as the constant A, depend on the tested biomaterial as indicated in Table 4.

From Eq. (1), the maximum tar yield is expected to decrease exponentially with temperature above 500°C, unlike with the relationship proposed by Samolada and Vasalos, in which the tar yield Y is expressed by the following polynomial form as a function of temperature:

$$Y = 55.19 - 11.5\,X - 21.69\,X^2$$

where $X = (T\text{-}560)/160$ is the dimensionless pyrolysis temperature.

In contact with the packing, the pyrolysis gas is subjected to thermal cracking. The kinetics of this thermal cracking is considered to be of a first order with respect to the remaining non-cracked part $X_g(T,t)$

$$\frac{dX_g(T,t)}{dt} = -kX_g(T,t) \tag{2}$$

By integrating Eq. (2), the tar yield after an exposure time t on the packing can thus be expressed as:

$$X_g(T,t) = X_g(T,0)\exp(-k.t) \tag{3}$$

The kinetics constant k obeys the Arrhenius law:

$$k = k_0\exp\left(\frac{-E}{R(T+273.15)}\right) \tag{4}$$

Following a simple trial-and-error procedure, the fit of the experimental curves of tar cracking led to the following set of optimal values for the pre-exponential factor k_0 and of the activation energy E, not depending on the raw biomass:wood, coconut shell or straw.

$$k_0 = 4.34\ s^{-1}$$

$$E = 23400\ \text{Jmole}^{-1}$$

Table 4
Constants for the kinetics of tar degradation

	A (1/°C)	X_g (500)
Wood	3.04×10^{-3}	0.35
Coconut shell	4.7×10^{-3}	0.24
Straw	3.5×10^{-3}	0.20

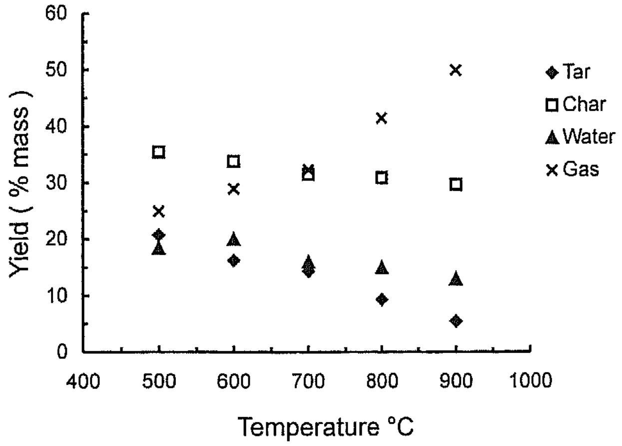

Fig. 7. Straw pyrolysis: effect of temperature on the decomposition products.

Fig. 8 shows a good agreement between experimental and calculated values for the tar yield versus time, with the optimal values of k_0 and E.

It is of interest to compare the kinetic parameters determined in the present work with others reported in the literature for cracking reactions of tar. These parameters are summarised in Table 5. All the parameters are available for wood as virgin material, however the nature of wood as well as the experimental processes are not the same for all the investigators. Except for the works of Chan et al., which brought on the radiative devolatilisation of thick pieces of wood, all the research works of the quoted investigators deal with flash or rapid pyrolysis of finely-ground wood, with nevertheless differences in the processes. Thus, Lédé [18] used a cyclone reactor

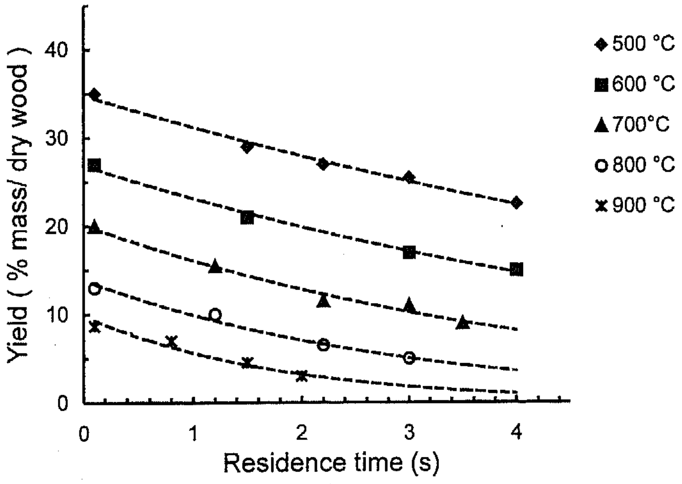

Fig. 8. Kinetics of tar cracking.

Table 5
Kinetics parameters for tar decomposition from various literature sources

Author	k_0 (s^{-1})	E (kJ/mole)
Chan et al. [10]	7.0×10^3	83.6
Kosstrin [8]	3.26×10^4	72.8
Liden et al. [12]	4.28×10^6	107.5
Diebold [11]	1.55×10^5	87.6
Boroson et al. [13]	9.53×10^4	93.3
Lédé [19]	5.9×10^7	123.48

while Kosstrin [8], Diebold [11] and Liden et al. [12], exploited the data obtained from a fluidised-bed technique. In all cases, both the reactions of solid and tar decomposition take place simultaneously in the same reactor — hence the accuracy of the residence times of volatile tar is questionable. Boroson et al. [13] have specifically studied the reaction of tar decomposition by using a set-up of two distinct reactors of small capacity, the first one devoted to solid pyrolysis, the second to tar cracking under a laminar flow of carrier gas (N_2), without packing.

From Table 5, it is obvious that the activation energies proposed by the cited authors, all above 72 kJ/mole, are significantly larger than that one evaluated in the present study. This essentially results from the different kinetic approaches. According to a first order decomposition rate, the tar yield shows an exponential decreasing with time from an initial value at time zero which is only a theoretical value of the maximum recoverable tar fraction, never experimentally measured, from most of the cited investigators. Thus Liden et al. have expressed the residual-tar yield ϕ as a function of residence time t_G and the maximum tar yield ϕ^* as

$$\Phi = \Phi^* \frac{(1 - \exp[-k_2 t_G])}{k_2 t_G}$$

The reported theoretical maximum tar yield varies from author to author. For Liden et al., it is equal to 0.703 while for Boroson et al. it is equal to about 0.52. Whatever the author, the maximum tar yield does not depend on the temperature at which the thermal degradation of tar is performed. By contrast, in the present study, the maximum tar yield, instantaneously generated at time zero, was found to decrease with temperature, according to Eq. (1).

With the kinetic parameters proposed in the present work, the predicted times for tar cracking at a given temperature are significantly longer than those obtained from the kinetic data due to Kosstrin et al., which are rather similar. Such a difference in the cracking times could be explained by differences in the cracking medium, i.e. when the cracking of tar takes place within a fluidized bed of sand, that involves an intimate contact between volatile products and the solid particles and consequently enhances the cracking rate. It can be easily estimated that the rate of cracking and thereby the attached kinetic parameters are specific to the nature and the granulometry of the cracking medium. The one used in the present work has the advantage of not inducing too large a pressure loss.

4. Conclusion

A quantitative evaluation of the different pyrolysis products from three ligno-cellulosic materials has been achieved. There is a difference in the molar fractions of gases generated as well as in the energy contents of the gas mixtures. The study extends the pyrolysis investigation to substances other than wood. The results can help in the design of multi-fuel gasifiers. The thermal cracking of tar has been studied, and a kinetic model proposed. This model quantifies the tar content of gas from a wood gasifier and allows the calculation of its heating value.

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