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DEVELOPMENT OF A PYROLYSIS TECHNOLOGY TO PRODUCE LARGE QUANTITIES OF CHARCOAL FOR THE IRON AND STEEL INDUSTRY[1]

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Abstract
Modern integrated iron and steelmaking processes utilising fossil carbon theoretically permit the substitution of 30% or more of coal or coke by charcoal without the need for substantial technological modification. However, in the majority of regions, the replacement of coal or coke by renewable carbon made from biomass (charcoal) is restricted by the limited availability and high price of charcoal. The development of an efficient pyrolysis technology capable of making large quantities of metallurgical charcoal from low-grade wood has the potential to improve this situation. A desirable new process also needs to be continuous with high level of automation, operatable in the reactors of broad size range, achieve high charcoal yield and be capable of capturing the value of by-products (bio-gas and bio-oil). A series of laboratory-scale experiments has been carried out under well-controlled conditions to identify the optimal process parameters for producing metallurgical charcoal from woody biomass at high yields. In this study the main process parameters were varied in a systematic manner and their effects on charcoal yield were quantified. The data obtained have enabled the judicious choice of a base concept for the development of an efficient process for continuous pyrolysis of biomass.
Key words: Integrated iron and steelmaking; Biomass pyrolysis; Charcoal; Process optimisation.

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1 INTRODUCTION

Replacement of fossil carbon (coal and coke) by charcoal is an efficient lower risk path to substantially reduce the net CO_2 emissions by the iron and steel industry. For example, an Australian integrated iron and steelmaking process could allow substitution of approximately 32 to 58% of coal or coke by charcoal without the need for substantial technological modification[1]. Minimal impact on the existing iron and steel making technology is expected if the replacement of fossil carbon by charcoal occurs within the following operations[1]:

- blast furnace tuyere injection of charcoal instead of pulverised coal;
- partial or complete substitution of coke breeze or anthracite used for iron ore sintering;
- replacement of nut coke charged into a blast furnace with the ferrous burden;
- making charcoal-based carbon/ore blast furnace pellets or pre-reduced feed to basic oxygen furnace;
- additions of charcoal to cokemaking blends.

However, in the majority of regions, the replacement of coal or coke by charcoal is restricted by its limited availability and high price. The affordability of charcoal would improve if it is made from low-cost feedstock, e.g. waste wood or wood residues, and if the efficiency of charcoal making technology is improved. It is broadly accepted that the attributes of an efficient technology are as follows:

- achieves high yields of main product (charcoal),
- has high productivity,
- has high energy efficiency,
- is based on a continuous process with a high level of automation,
- is operatable with reactors of broad size range,
- captures the valuable by-products.

Pyrolysis of wood is a commonly used technology to make charcoal. In most general terms pyrolysis can be defined as thermal decomposition in an oxygen-free or oxygen-poor atmosphere and at temperatures above 300°C. The by-products of charcoal making are condensables (liquids) and gases. Pyrolysis processes can be broadly divided into slow pyrolysis, when the heating rate of material is typically lower than 20°C/min, and fast pyrolysis with higher heating rates exceeding 100°C/s[2]. The yield of charcoal from wood in fast pyrolysis is approximately one third of that in slow pyrolysis, therefore slow pyrolysis is a preferred process for charcoal making.

Among the existing slow pyrolysis technologies only a few can process low-grade wood (e.g. wood waste or wood residues) in a continuous manner. These are a multiheath furnace[3], a rotary furnace[4] and a furnace based on an Auger reactor (material is driven and stirred by a screw)[5]. The main technological challenge that needs to be overcome to enable the pyrolysis of low-grade wood is heating large masses of small-sized wood to the required temperature. Small-sized wood has very limited gas permeability; therefore, efficient heating by the flow of hot gas through the bed of material is not possible. Additionally, low thermal conductivity of wood restricts the efficiency of heating the material externally. As a result, pyrolysis reactors for small-sized wood are limited in scale and need a means to agitate the mass of material, in order to accelerate the delivery of heat to its core. The agitation of material is enabled by moving parts inside the high-temperature zone of the reactors, which increases their mechanical complexity. These drawbacks of the existing technologies lead to an increase in installation and maintenance costs of a pyrolysis plant, and impact on the price of the final product.

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Internal combustion of a fraction of pyrolysis gases and vapours inside the reaction chamber is also practiced[3]. This may improve heating of the material, but not to the extent that the need for agitation can be eliminated. Additionally, the introduction of air into the reactor chamber results in the burn out of not only the volatile pyrolysis products but also some of the charcoal. Gaseous products of combustion dilute the pyrolysis gases and reduce their calorific value. Therefore, the development of an efficient pyrolysis technology capable of making large quantities of metallurgical-quality charcoal from low-grade wood is needed.

The development and optimisation of a new technology requires reliable information on the effect of the main process parameters on the yields and quality of products (charcoal, condensable products and gas). The available literature data suggest that apart from the maximum pyrolysis temperature and the nature of the feedstock, the following process parameters have significant impact on the yield and quality of pyrolysis products[6]:

1. heating rate of material;
2. size of particles/pieces of material, particularly for small-sized wood;
3. residence time of pyrolytic vapours/gases within the material bed, which can be described by the depth of bed material and by the rate of removal of the products from the reactor by the gas purging the reactor.

Although some information on the effect of these factors does exist, to the authors' knowledge no systematic study has been conducted that investigated the effect of these parameters for a single material. At the same time, the variability of the properties of various materials makes it difficult to compare the relative importance of these factors, if obtained in different studies and for different materials.

Therefore, this work is focussed on obtaining quantitative information on the effect of the main parameters of the pyrolysis process on its performance, which was characterised by the yield of the main product – charcoal. A single material with uniform properties was used throughout the study. The result of the investigation was used to choose a concept for a pyrolysis technology, which would be more efficient for the carbonisation of small-sized wood than the existing technologies.

2 MATERIALS AND METHODS

An Experimental Design approach was used to systematically characterise the effect of process parameters on the yield of charcoal. This approach provides comprehensive information on the effect of the parameters and their interactions, i.e. their mutual interdependence[7]. According to Di Blasi[6] the process parameters having a primarily influence on the yield of charcoal are as follows:

- the heating rate (HR)
- the flow rate of gas purging the bed of material (FR)
- the size of wood particles (PS)
- the depth of the bed of material (BD).

These main process parameters ("factors" in accordance with Experimental Design terminology[7]) were chosen as independent variables and the dependent variable was the yield of charcoal (ChY). To obtain complete information on the mutual interactions of the process parameters it is necessary to run the experiments in accordance with a so-called full factorial plan, when the response of the system is obtained for all the possible combinations of the process parameters. For our case of 4 independent variables (factors) and when every variable takes 2 values (levels) a

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minimum of 16 experiments need to be conducted. According to the terminology common for Experimental Design this is called a 4^2 full factorial plan[7].
The high and low levels of the independent variables (factors) are shown in Table 1. For all the factors except for BD these levels of variation were defined by practical considerations for pyrolysis technology. The maximum BD, which could be studied, was limited by the height of the isothermal hot zone of the laboratory furnace used.

Table 1. The levels of variation of the independent variables

Independent variable (process parameter)	HR, °C/min	FR*, L/cm²min	PS, mm	BD, cm
Higher level	16	0.41	21	19
Lower level	5	0	7.2	7

** The flow rate of gas through the unit area (1 cm²) of the cross-section of an empty crucible*

A standard full factorial 4^2 plan of the experiments[7], when applied to our study, can be represented by the matrix shown in Table 2, where "+" denotes the higher level of a factor, and "–" denotes the lower level of a factor.
Pinus Radiata from a local supplier (Victoria, Australia) was chosen as the biomass material for this work. To maintain uniformity of the material properties, a single 6 m long beam of kiln dried wood was used as the source of biomass for all the experiments conducted in this work. The beam was cut from the sapwood of the original tree. It was inspected for defects and those parts of the beam that contained knots or compression wood were cut off and excluded from use. The remaining parts of the beam, uniform in their appearance, were sawn across the principal grain axes into 5 mm and 21 mm thick slices. The 5mm slices were crushed into irregular fractions and then sieved. The sieved fraction (-8.0 mm +6.3 mm) was used for the experiments. To average any variations in the properties of the wood particles, the entire amount of the material prepared was placed in a drum and stirred before use. The 21 mm slices were cut into regular cubic pieces 21x21x21 mm and also were mixed to average any minor variations in their properties before being used in the experiments. Wood particles prepared in this manner were dried in an oven at 103°C for at least 24 hours and then loaded into a crucible immediately before the beginning of the experiment.

Table 2. The matrix of a standard full factorial 4^2 plan used for the experiments

Experiment No	FR	HR	PS	BD
1	+	+	+	–
2	+	+	–	–
3	+	–	+	–
4	+	–	–	–
5	–	+	+	–
6	–	+	–	–
7	–	–	+	–
8	–	–	–	–
9	+	+	+	+
10	+	+	–	+
11	+	–	+	+
12	+	–	–	+
13	–	+	+	+
14	–	+	–	+
15	–	–	+	+
16	–	–	–	+

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Experiments were conducted in a vertical ceramic tube furnace with the internal diameter of the tube being 50 mm. Glow bar heating elements were mounted outside of the tube. The tube was sealed at its both ends, and purging it with high-purity nitrogen permitted the maintenance of the oxygen-free environment inside the tube. A wood sample was placed in a stainless steel cylindrical crucible with an internal diameter of 36 mm. The amount of wood in the crucible was adjusted so that the depth of the material bed was 7 cm, which corresponded to a sample mass of 14 g approximately. The bottom of the crucible and its lid were made from stainless steel 2x2 mm mesh, which permitted sweeping the sample with nitrogen at controlled rate. The direction of the nitrogen flow through the bed material was from its top towards the bottom. Two K-type thermocouples were submerged in sample in such a way that their hot ends were positioned at 2.3 and 4.6 cm above the bottom of the crucible. This enabled control of the temperature of wood during pyrolysis.
For the experiments with a deeper bed of material, another cylindrical crucible with the same diameter was attached to the top of the main crucible. The depth of the material bed in the top crucible was 12 cm, which resulted in the total bed depth of wood particles of 19 cm. The bottom crucible was separated from the top crucible by stainless steel mesh assuring that material in the bottom crucible was not mixed with the material from the top crucible. The yield of charcoal from wood placed in the bottom crucible was recorded for these experiments. For the experiments with no flow of nitrogen through the bed of wood, the top of the main crucible (for the experiments with BD=7 cm) or the top of the top crucible (for the experiments with BD=19cm) was closed by an air-tight lid, therefore the outflow of pyrolysis vapours and gases could occur through the bottom of the main crucible only. The material in the top crucible was effectively a generator of pyrolysis vapours sweeping through the material placed in the bottom crucible.
To provide some assurance that the limitation of the maximum bed depth to 19 cm did not introduce a considerable error in the evaluation of the effects of this process parameter on the yield of charcoal, some experiments with expanded (larger diameter) top crucible were also conducted. The expanded crucible contained 60% more wood than the 36 mm diameter top crucible. The reason of using an expanded diameter crucible was to model a deeper bed of material (greater than 19 mm), while keeping the total height of the assembly of crucibles within the height of the isothermal zone of the furnace, which was 20 cm. Since the whole batch of material placed in the expanded crucible was located within the isothermal zone of the furnace, and all the vapours generated in that crucible were directed downwards into the bottom crucible, it was assumed that the effect of the expanded crucible was the same as it would have been for ~60% higher uniform diameter crucible.
Since the experiments with the expanded top crucible demonstrated that the extension of BD beyond 19 cm did not result in any measurable effect on charcoal yield, it was concluded that the BD of 19 cm chosen for the mainstream testwork was acceptable.
For all the experiments the maximum temperature of pyrolyisis was 650°C. The yield of charcoal was determined gravimetrically using a Sartorius (extended series GK1403) three decimal place precision balance.

3 RESULTS AND DISCUSSION

The experimental data collected in accordance with the plan shown in Table 2 are summarised in Table 3 below. The analysis of the data obtained was performed

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using a standard procedure for full factorial plans of experiments and is described in detail elsewhere[7]. This analysis permitted the description of the effects of the main process parameters on the yield of charcoal in the form of the following correlating equation:

$$ChY = b_0 + b_1 \cdot fr + b_2 \cdot hr + b_3 \cdot ps + b_4 \cdot bd + b_{1,2} \cdot fr \cdot hr + b_{1,3} \cdot fr \cdot ps + b_{1,4} \cdot fr \cdot bd + b_{2,3} \cdot hr \cdot ps + b_{2,4} \cdot hr \cdot bd + b_{3,4} \cdot ps \cdot bd \quad (1)$$

Where ChY – is the yield of charcoal (% of dry wood weight); $b_0, b_1, \ldots, b_{34}$ are the regression coefficients; fr, hr, ps and bd are the relevant non-dimensional factors expressed as:

$$fr = \frac{\mathrm{FR}[\mathrm{L/cm^2 min}] - 0.205\,\mathrm{L/cm^2 min}}{0.205\,\mathrm{L/cm^2 min}} \quad (2)$$

$$hr = \frac{\mathrm{HR}[^\circ\mathrm{C/min}] - 10.5\,^\circ\mathrm{C/min}}{5.5\,^\circ\mathrm{C/min}} \quad (3)$$

$$ps = \frac{\mathrm{PS[mm]} - 14.1\,\mathrm{mm}}{6.9\,\mathrm{mm}} \quad (4)$$

$$bd = \frac{\mathrm{BD[cm]} - 13\,\mathrm{cm}}{6\,\mathrm{cm}} \quad (5)$$

Table 3. Results of the tests conducted in accordance with 4^2 full factorial plan of experiments

Experiment No	Yield of Charcoal, % of dry wood weight	FR, L/cm^{2}min	HR, $^\circ$C/min	PS, mm	BD, cm
1	22.51	0.41	16	21	7
2	20.44	0.41	16	7.2	7
3	24.23	0.41	5	21	7
4	20.70	0.41	5	7.2	7
5	22.31	0	16	21	7
6	22.44	0	16	7.2	7
7	24.82	0	5	21	7
8	24.86	0	5	7.2	7
9	22.69	0.41	16	21	19
10	21.08	0.41	16	7.2	19
11	23.98	0.41	5	21	19
12	21.91	0.41	5	7.2	19
13	22.58	0	16	21	19
14	23.00	0	16	7.2	19
15	25.49	0	5	21	19
16	25.48	0	5	7.2	19

The regression coefficients obtained from the experimental results are provided in Table 4.

Table 4. Regression coefficients of equation (1)

b_0	b_1	b_2	b_3	b_4	$b_{1,2}$	$b_{1,3}$	$b_{1,4}$	$b_{2,3}$	$b_{2,4}$	$b_{3,4}$
22.961	-0.840	-0.901	0.544	0.244	0.389	0.616	-0.021	-0.153	-0.038	-0.135

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Therefore the equation correlating the yields of charcoal as a function of the main process parameters takes the following form (the regression coefficients $b_{1,4}$ and $b_{2,4}$ are insignificant):

$$ChY\ [\text{\% of dry wood weight}] = 22.961 - 0.84 \cdot fr - 0.901 \cdot hr + 0.544 \cdot ps + 0.244 \cdot bd + 0.389 \cdot fr \cdot hr + 0.616 \cdot fr \cdot ps - 0.153 \cdot hr \cdot ps - 0.135 \cdot ps \cdot bd \quad (6)$$

Where fr, hr, ps and bd are defined by eqs. (2) to (5).
The regression coefficients of this equation are depicted in Figure 1. Since eq. (6) is written for non-dimensional factors, the values of the regression coefficients show the relative significance of each factor (process parameter) as well as the significance of the interactions between the factors. Negative values of the regression coefficients mean that the increase in a certain factor will result in a reduction of charcoal yield.

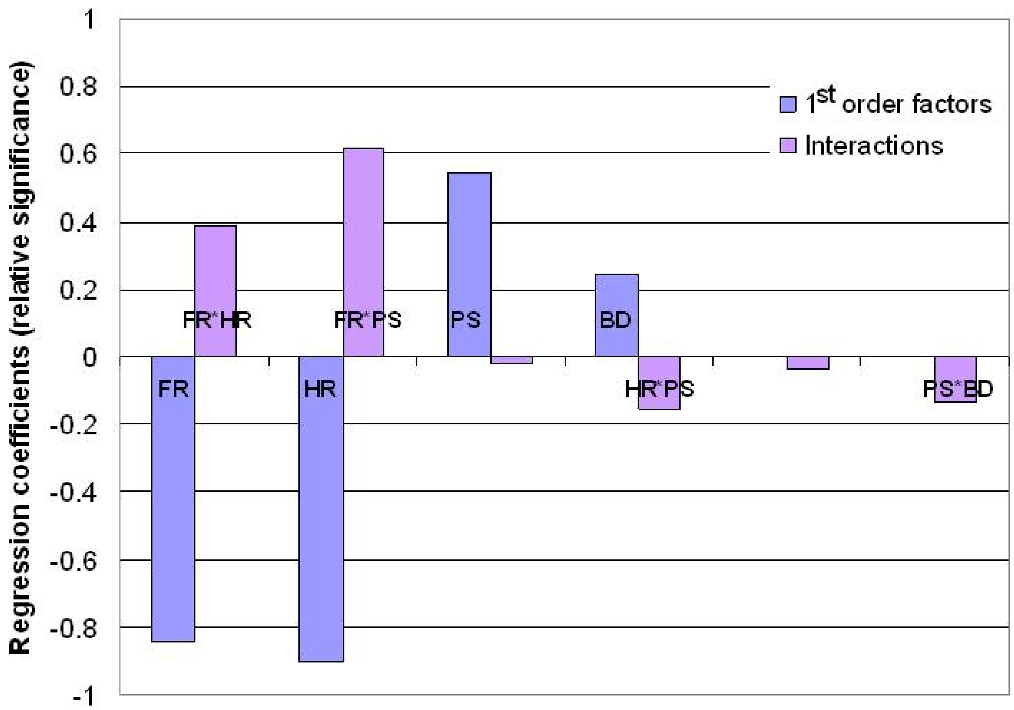

Figure 1. Illustration of the relevant significance of four main process parameters (FR, HR, PS and BD) and of their interactions in terms of their effect on the yield of charcoal.

In accordance with this chart the influence of the main process parameters on the yield of charcoal decreases in the following sequence: (1) heating rate; (2) flow rate of gas through the sample; (3) the size of particles; (4) the bed depth.
It has also been revealed that there are three strong mutual interactions between the parameters of the system. These are:

- The interaction between the FR and PS: the effect of the particle size on the charcoal yield depends substantially on the flow of gas through the sample.
- The interaction between the FR and HR: the effect of the heating rate depends substantially on the flow rate of gas through the sample and vice versa.
- The interaction between the HR and the PS: the effect of the heating rate depends on the size of wood particles.

The results of the experiments summarised in Table 3 can be presented graphically only if they are subdivided into smaller fractions. As an example, the data for no flow and for high flow of nitrogen through the sample with BD = 7 cm are plotted in

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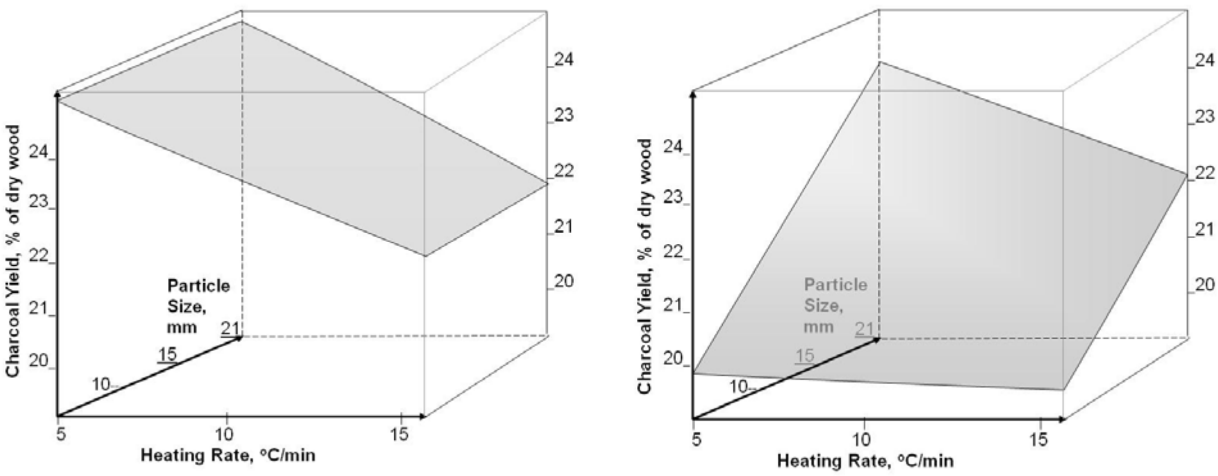
Figures 2a and 2b. These 3-dimesional plots illustrate the interactions between HR, PS and FR. At no flow of nitrogen (Fig. 2a) the yield of charcoal becomes independent of the particle size. At the same time, at the high flow of nitrogen (Fig 2b) the size of particles has a significant influence on the yield of charcoal. With the increase of the flow rate of purging nitrogen the effect of heating rate diminishes, particularly significantly for small wood particles. At FR = 0.41 L/cm^{2}min for smaller particles, the variation of the heating rate has almost no effect on the yield of charcoal, while for larger particles the effect of HR is strong.



a b

Figure 2. Combined effects of the heating rate and the particle size on the charcoal yield for BD = 7 cm. (a) no flow of nitrogen through the bed of wood particles; (b) a flow of nitrogen through the bed of wood particles of 0.41 L/cm^{2}min.

It worth noting that a decrease of either FR or HR, or an increase of PS result in the extension of the residence time of vaporous and gaseous pyrolysis products within the sample of wood. Therefore, this supports a broadly accepted fact, that an increase in the residence time of pyrolysis vapours and gases is a significant factor acting towards an increase in the charcoal yield[6].

4 CHOICE OF PYROLYSIS TECHNOLOGY

The experimental work described above makes it possible to identify the most significant features of a pyrolysis process designed to maximise the yield of the main product – charcoal. These features are as follows:

1. low heating rate of wood (it needs to be noted, that a too low heating rate would negatively impacts on the productivity of a pyrolysis plant, hence an optimal heating rate needs to be found);
2. minimal flow rate of gas purging the bed of material;
3. reasonably large size of wood particles, if purging the material bed by gas cannot be avoided; however, at no flow of gas the particle size has insignificant effect on the yield of charcoal.

An important parameter, which is required for the design and optimisation of a pyrolysis process is the heat of pyrolysis (ΔH_{pyro}), as this is directly linked to the heat balance of the process. Unfortunately, it appears that ΔH_{pyro} cannot be explicitly defined from the information available in the literature. The known data on the heat of

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the process vary from +2,510 J/g* (overall endothermic) to –2,100 J/g (overall exothermic)[8]. However, an important trend has been noticed: ΔH_{pyro} is directly proportional to the amount of charcoal generated as a result of wood pyrolysis[8]. Since the yield of charcoal increases with an increase in the particle size of wood, the data obtained with larger particles or with larger batches of material may provide a better representation of the process occurring in a larger-scale reactor. Although such data are scarce they make it evident that the heat of pyrolysis for larger samples of wood tends to be substantially exothermic and may comprise up to 10% of the calorific value of wood [9-10]. Therefore, although an accurate value of ΔH_{pyro} is not currently known, it can be concluded that efforts towards an increase in charcoal yield may results in a substantial increase in heat generated by the process to such an extent, that it may cover a considerable fraction of the energy needs (if not all the needs) of the process.

Other factors to be taken into consideration for an efficient modern pyrolysis process are as follows:

- the process to be continuous with high level of automation,
- the process to be operatable with reactors of a broad size range,
- the process to be capable of capturing the value of by-products (bio-gas and bio-oil).

Despite processing small-sized wood presents a significant technological challenge, conducting pyrolysis of such a wood attracts considerable benefits. Using small-sized wood as a feedstock would substantially lower the cost of feed material (by incorporating wastes, residues or pellets made from them) and also has a potential to reduce the residence time of materials in the reactor, if required.

After considering the available information on the existing pyrolysis technologies or those that did exist in the past, which are capable of processing small-sized wood in a continuous manner, it was concluded that a process based on a vertical-shaft reactor and efficiently utilising the exothermic effects of pyrolysis is the best suited prototype for the development of a new technology. In such a process wood is reduced in size, well dried and loaded into a reactor from its top. Heating of material to the carbonisation temperature occurs by heat transfer from ascending hot pyrolysis vapours and gases generated in the reaction zone near the middle of the shaft. Pyrolysis is conducted in an oxygen-free atmosphere and the reactor is sealed to avoid the ingress of air. A distinctive feature of the process is that it may not need any external or internal source of heat to maintain the required temperature in the reaction zone and the only sources of heat are the exothermic reactions accompanying the thermal decomposition of wood in an oxygen-fee environment. The reactor should be well insulated to minimise heat losses. As the main source of heat is located within the bulk of the material itself, this process is free from the need to deliver heat to the material from an external source or via purging hot gas through the bed of material. Due to these features, such a process is well suited for processing small-sized wood (which is usually difficult due to low thermal conductivity and insufficient gas permeability of such a wood) and is expected to have a unique ability to work in reactor units of a very broad range of sizes (as no delivery of heat from the exterior of the reactor is need to maintain the required temperature).

* *Heat of pyrolysis is expressed on a per unit mass of volatile basis (volatile materials generated during wood pyrolysis)*

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Therefore, a new process based on this concept would closely comply with the requirements for an efficient pyrolysis process as described above. Namely:

1. can process small-sized wood;
2. no purging of material by gas is needed, which maximises the yield of charcoal and ensures that pyrolysis gases are not diluted by a non-combustible gas (pyrolysis gas retains its maximum calorific value);
3. the condensable pyrolysis products are not excessively diluted by moisture as well-dried wood is used as a feedstock (condensable pyrolysis products retain high value);
4. exceptional scaling-up ability, as no external heating of material is needed;
5. no burn out of pyrolysis products as the thermal decomposition of wood occurs in an oxygen free environment;
6. high energy efficiency, as no external source of high-temperature heat is needed to maintain the required temperature in the reaction zone of the reactor and only low-grade heat is required to dry the feedstock;
7. low capital and maintenance costs as the reactor is mechanically simple and has no moving parts within its high-temperature zone.

5 CONCLUSIONS

The effects of the flow rate (FR), the heating rate (HR), the particle size (PS) and the depth of the bed of material (BD) have been studied using Experimental Design. The relative significance of four main process parameters and their interactions has been revealed. HR, FR, PS and BD all have a significant influence on the yield of charcoal with HR and FR being most significant. The interactions between FR and PS, and between FR and HR are the most important interactions.
As the base concept for the development of a new pyrolysis technology the principle of an autogenous pyrolysis was chosen – a process which is capable of thermally decomposing and carbonising wood in an oxygen-free atmosphere without the need to supply an external heat to the reactor.

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