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## **Production of charcoal from different wood species**

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### **Abstract**

The study presented hereby is the first part of a more general investigation concerning the valorization of wood wastes by activation of carbon obtained by pyrolysis of different wood species. 6 different European wood species have been tested and their ability to produce charcoal has been qualified. Different series of tests on cubic wood samples have permitted to determine the dimensions variations and mass losses induced by the thermal degradation of wood during its carbonization. In particular, the conversion ratio of wood material into charcoal has been determined as a function the wood species and the pyrolysis conditions. The experimental results show that the carbonization is complete for all the species from a temperature of 500 °C and that the conversion ratio of the anhydrous mass of wood into carbon does not depend on the wood species studied.

**Key words :** wood, pyrolysis, conversion ratio, wood waste valorization

### **1. Introduction**

Wood waste valorization has become an up to date subject of research mainly because our modern society feels more concerned by the optimization of using natural resources. Many solutions have been proposed but we can distinguish four major ones. The first solution consists in an energetic valorization of wood wastes by combustion. It is particularly interesting if wood wastes do not contain a too high proportion of chemical additives the thermal degradation and oxidation of which could produce toxic compounds. In this case, the treatment of the gas phase obtained after combustion could be expensive and decrease the benefit of such a valorization.

A second possible valorization is the gasification. Some interesting studies have been presented by Chen and al. (1997), Iniesta and al. (2001), Ollero and al. (2003) or Struis and al. (2002). The kinetics of gasification is described for different biomasses according to different processes. Wood wastes are thermally degraded under a neutral atmosphere in order to produce hydrogen or methane. This solution is also interesting if the initial wood wastes are not too polluted by some chemical compounds the degradation of which could generate toxic fumes.

The third solution consists in the production of new materials such as particles boards or fibers boards but is not possible for all wood species wastes since some of them contain natural compounds which are not compatible with particles gluing.

These three first solutions are now well known but a more interesting solution, providing a higher commercial value to the final product, can be envisaged: carbonization of wood wastes in order to produce activated carbon. The aim of the production of activated carbon is to transform a material considered as a waste (such as biomass wastes, oil petrol residue...) into a high added value product since the average price of activated carbon in Europe is between 600 and 800 euros/m<sup>3</sup>.

Many data are available on the production of activated carbon. Pulido-Novicio and al. (2001) have characterized the adsorption capacities of some wood charcoal. Von Blucher and De Ruiter (1999) have proposed a new process to produce granulated activated carbon. The contribution of Young (1996) confirms that many agricultural commodities are convenient for the production of activated carbon.

The demand for higher purity products in many fields, and especially in the life science industry, is getting stronger and stronger. Adsorption processes are powerful tools to address these purity demands. It must also be mentioned that for several reasons (environment, safety, costs...), many extraction processes involving large quantities of organic solvents are nowadays replaced, partially or totally, by solid phase adsorption processes. Activated carbon is certainly the most famous and practical absorbent in particular because its range of use is very large and concerns many pollutants. The quality of activated carbon (and therefore its price) is a function of the specific area characterizing the mass exchange between carbon particles and the fluid to depollute. This exchange area, according to Burdock (1997) can vary between some 100 m<sup>2</sup> per g up to more than 2000 m<sup>2</sup>/g. An other important physical characteristic is the size pores distribution: according to its use, macro-pores (the pore radius of which is some hundreds μm) or micro-pores (the pore radius of which is some ηm) can be preferred. Moreover, all the depollution processes do not necessarily need high quality activated carbon. These are the reasons why it could be interesting to study the ability of different biomass wastes to produce charcoal and to determine the characteristics of this charcoal.

The investigation presented in this paper proposes to test 6 wood species having very different initial characteristics. These 6 different wood species are : ash (*Fraxinus excelsior*), beech (*Fagus sylvatica*), douglas (*Pseudotsuga menziesii*), oak (*Quercus robur*), white poplar (*Populus alba*) and sitka spruce (*Picea sitchensis*). The operating parameters of their pyrolysis have been studied. Some properties of the final product are presented permitting to suggest some perspectives for the charcoal produced by pyrolysis.

## 2. Experimental device and protocol

### 2.1 Experimental device

Different series of pyrolysis have been performed on a laboratory scale pyrolyser the scheme of which is presented by Figure 1.

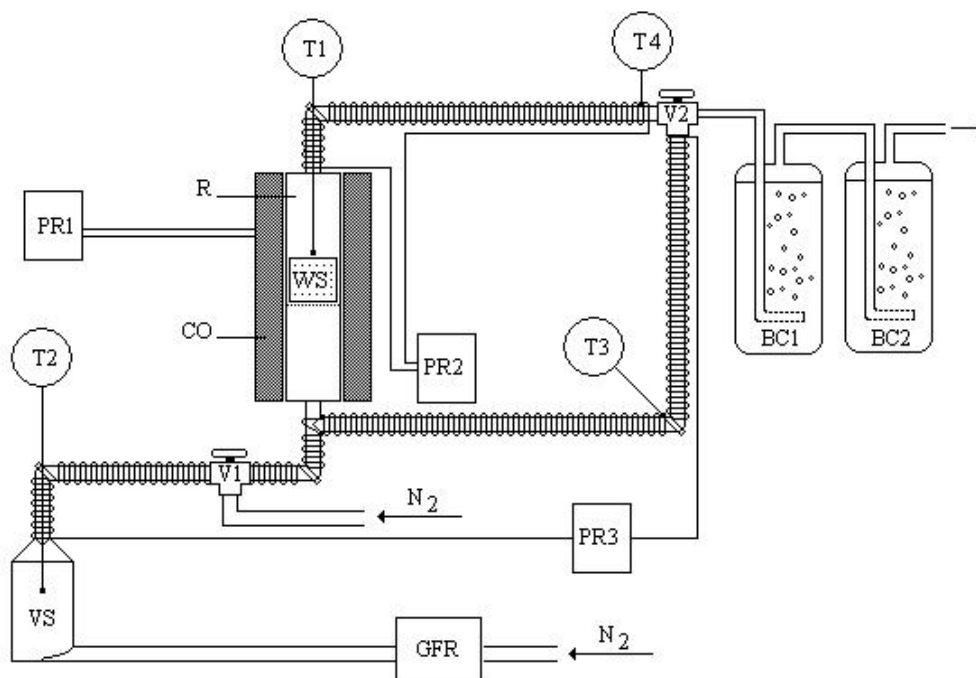


Figure 1: Scheme of the experimental device

This pyrolyser is constituted by a stainless steel tube which inside diameter is 50 mm. The length of this tube (that we will be called “reactor”) is 60 cm but only the central part, where the temperature profile is constant, is used for pyrolysing wood waste samples (WS). This reactor (R) is inserted into a cylindrical

oven (CO) connected to a power regulation (PR1) permitting to heat up to 1000 °C. It can be crossed by a gas (nitrogen) the flow rate of which can be fixed at a constant value with a gas flow rate regulator (GFR). The sample to pyrolyse is deposited inside a metal basket which takes place on a grid. This technical solution allows to withdraw after carbonization all the solid residue without any loss.

The inlet gas flow rate and the pyrolysis exhaust fumes circulate in ¼ inch stainless steel tubes the temperature of which is maintained constant by spiral electrical resistances connected to two power regulations (PR2 and PR3). The inlet gas flow can cross a saturator (VS) the function of which is to generate vapor (for example water vapor) if necessary. The exhaust fumes produced by pyrolysis are sent to a double bubbling chamber (BC1 and BC2) in which the tars, in particular, can be condensed.

It is possible to make the installation work in closed loop by adjusting the position of two valves (V1 and V2).

All the installation is equipped with several thermocouples (T1 to T4) permitting to follow the temperature variations of the device at different positions and can be easily dismantled for cleaning operation after some pyrolysis tests.

## 2.2 Experimental protocol

Six different wood species have been selected for our study mainly because their wastes are produced by wood industry at a large scale and have a low economical value induced by some strong defects (wood discoloration, knots or low mechanical resistance for example). Two softwood species (douglas and spruce) and four hardwood species (beech, poplar, oak and ash) have been pyrolysed at fixed temperature from 400 up to 700 °C.

The initial physical characteristics of these wood species are very heterogeneous: some have very large grown rings (spruce for example) or very thin (oak), some have a high density (beech) while some others have a very low density (poplar). For these reasons, it is interesting to evaluate the influence of these initial physical characteristics on the final product (charcoal).

All the samples are cut from air dried wood pieces the initial moisture content of whose is about 6 %. For each tests of pyrolysis, we prepare 10 cubes, the edge of which is 1.50 cm ( $\pm 0.02$  cm). The three wood directions (longitudinal, radial and tangential) can be easily detected: the cubes are cut so that the growth rings are well parallel to two opposite faces (see Figure 2). Their initial mass  $m_i$  (g) and all initial dimensions are measured with precision tools ( $10^{-4}$  g balance and electronic caliper). Then they are placed in an oven at 105 °C ( $\pm 2^\circ\text{C}$ ) in order to determine their oven dry mass  $m_0$  (g) and to deduce their initial wood moisture content  $x_i$  (%) by the following relation :

$$x_i = 100 \cdot \frac{m_i - m_0}{m_0} \quad (1)$$

Some initial physical characteristics of the samples are listed on table 1 and show that the wood species chosen are very different from each others.

*Table 1. Initial physical characteristics of the samples*

Wood species	Average initial oven dry density (g/cm <sup>3</sup> )	Average dimension of the growth rings (mm)	Visible defects
Ash	0.53 – 0.66	1.0 – 1.5	/
Beech	0.45 – 0.60	2.0 – 2.5	/
Douglas	0.43 – 0.58	4.0 – 5.0	Large knots
Oak	0.55 – 0.68	1.0 – 1.5	/
Poplar	0.29 – 0.35	5.0 – 7.0	Small knots
Spruce	0.33 – 0.36	6.0 – 8.0	Large knots

Because each sample must be clearly identified before and after pyrolysis, they are all marked with a light pencil the inscription of which is still visible after pyrolysis (in fact the print of the pencil mark is visible). This easy way will permit to measure the size variations for all the dimensions of our samples induced by the carbonization.

The 10 wood samples are placed inside the reactor of pyrolysis. The device is hermetically closed and the nitrogen flow rate crossing the reactor is fixed at 2 L/min. A first drying step at 105 °C is used to be sure that all the samples are totally anhydrous before pyrolysis. Even if all the samples have been already oven dried, they may have absorbed some water vapor during their manipulation (weighting and dimensions measurements). This first drying step is also used to evacuate any trace of oxygen. After 1 hour, the reactor temperature is changed to reach its setting value (temperature of pyrolysis). The heating rate is then about 5°C/min. When this setting temperature is reached, we start to measure the pyrolysis time. The heating is stopped when this time is equal to the desired duration of pyrolysis and the reactor is slowly cooled. The carbonized samples can be withdrawn from the reactor when the inside temperature is about 150 °C.

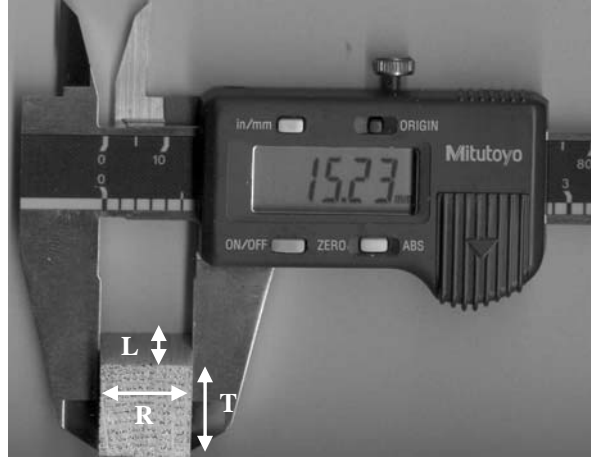


Figure 2. Picture of a sample (ash) showing the three wood directions  
 ( $L$  = longitudinal,  $R$  = radial,  $T$  = tangential)

One more time, the samples are weighted and their dimensions are measured. In particular, the relative mass loss  $M_L$  is deduced according to the following relation:

$$M_L = 100 \cdot \frac{m_{0i} - m_C}{m_{0i}} \quad (2)$$

where  $m_{0i}$  is the initial oven dry mass of a sample (g) and  $m_C$  is the mass of the same sample after pyrolysis (g).

We can also deduce the relative size variation coefficients  $R_L$ ,  $R_R$  and  $R_T$  (%) in the three directions (longitudinal, radial and tangential):

$$R_L = 100 \cdot \frac{l_{L0i} - l_{LC}}{l_{L0i}} \quad (3)$$

$$R_R = 100 \cdot \frac{l_{R0i} - l_{RC}}{l_{R0i}} \quad (4)$$

$$R_T = 100 \cdot \frac{l_{T0i} - l_{TC}}{l_{T0i}} \quad (5)$$

In relations (3) to (5),  $l_{X0i}$  is the initial oven dry dimension (cm) in the direction  $X$  ( $X = L, R$  or  $T$ ) and  $l_{XC}$  is the final dimension (cm) after carbonization in the direction  $X$  (these dimensions are used if the cubic form of carbonized samples is not conserved).

Finally, the initial oven dry density  $\rho_{0i}$  ( $\text{g}/\text{cm}^3$ ), the density of carbonized wood  $\rho_C$  ( $\text{g}/\text{cm}^3$ ), the relative density variation  $\alpha$  (%) and the theoretical porosity  $\varepsilon$  (%) can be calculated:

$$\rho_{0i} = \frac{m_{0i}}{l_{R0i} \cdot l_{T0i} \cdot l_{L0i}} \quad (6)$$

$$\rho_C = \frac{m_C}{l_{RC} \cdot l_{TC} \cdot l_{LC}} \quad (7)$$

$$\alpha = 100 \cdot \frac{\rho_{0i} - \rho_C}{\rho_{0i}} \quad (8)$$

$$\varepsilon = 100 \cdot \left( 1 - \frac{\rho_C}{\rho_{CG}} \right) \quad (9)$$

The previous relation is obtained by supposing that the final residue is only composed by graphitic carbon the density of which is  $\rho_{GC} = 2.25 \text{ g/cm}^3$  (the mass of wood minerals contained by this solid residue is neglected).

28 pyrolysis tests have been performed at different constant temperatures (from 390 to 672 °C) during 2 hours and an other 8 tests have been performed at 2 different temperatures (390 °C and 440 °C) with 4 different wood species (oak, poplar, spruce and Douglas) with a pyrolysis duration of 1 hour.

### 3. Experimental results and discussion

The first experimental results show that the relative mass loss after 1 hour of pyrolysis is equal to the one after 2 hours. This means that, according to our operating conditions (in particular to the dimensions of our samples), the maximum conversion at a fixed temperature is obtained after only 1 hour and that it is also the case after 2 hours.

On Figure 3 is represented the variation of the average relative mass loss  $M_L$  (of ten samples used for a same test of carbonization) as a function of the temperature of pyrolysis. We can observe that this parameter does not really depend on the wood species carbonized and that it tends to an asymptotic value near 75 % for temperatures higher than 500 °C.

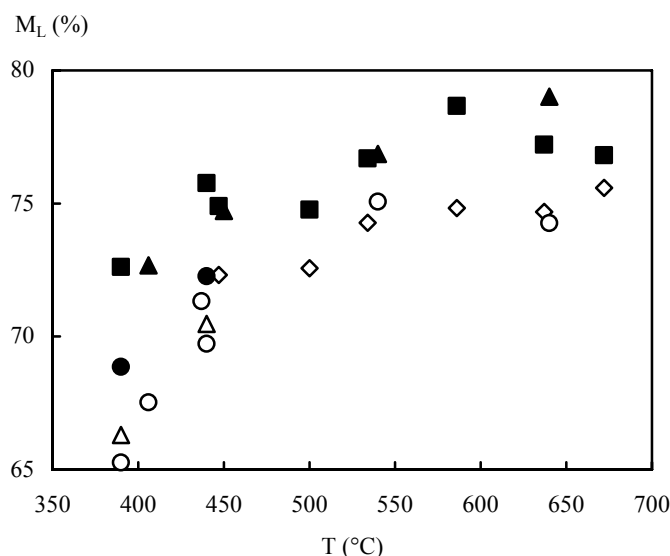


Figure 3. Variation of  $M_L$  as a function of the pyrolysis temperature for the 6 different wood species studied (◇ : Ash, ○ : Douglas, ● : Spruce, ■ : Poplar, △ : Oak)

Moreover, if we represent the maximum values of  $M_L$  (asymptotic value of  $M_L$  given by Figure 3 for each wood species) as a function of the initial oven dry density (Figure 4), we can see that there is absolutely no correlation between this parameter and the initial physical property represented by the initial oven dry density. The slight differences those can be observed may be due to some experimental variations such as, for example, the disposition of the samples (provision in bulk) in the reactor during the pyrolysis or a slight variation of the nitrogen flow rate (inducing a slight change of the heat exchange coefficient between the samples and the gas phase).

There is also no correlation between the different size variation coefficients and the initial physical characteristics: Figure 5 shows that, for the 6 wood species, the average size variation coefficient (of ten samples used for the same test of carbonization) only depends on the temperature of pyrolysis.

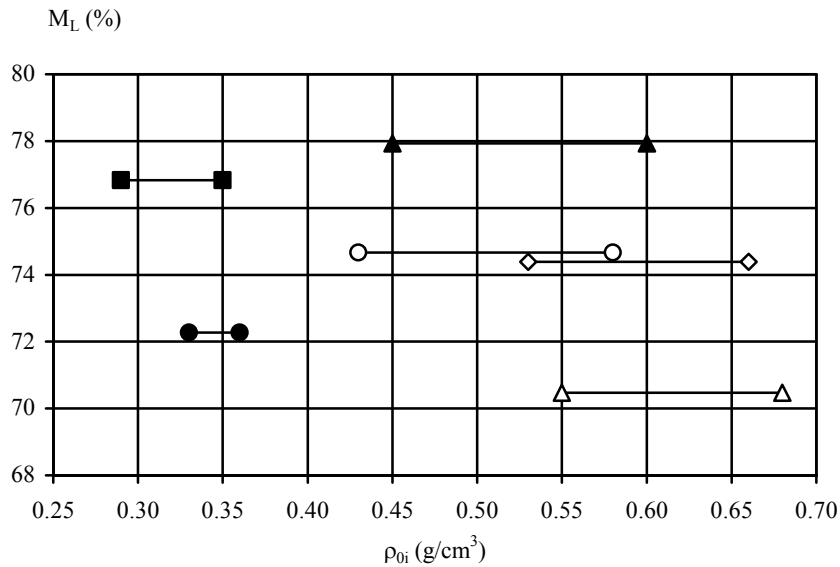


Figure 4. Variation of the relative mass loss as a function of the initial oven dry density of the wood samples ( $\diamond$  : Ash,  $\circ$  : Douglas,  $\bullet$  : Spruce,  $\blacksquare$  : Poplar,  $\Delta$  : Oak)

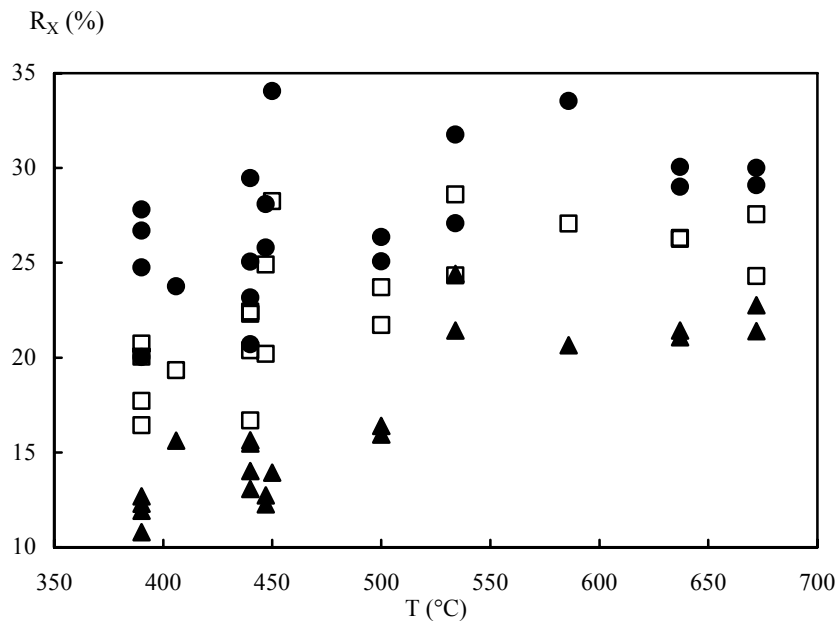


Figure 5. Influence of the pyrolysis temperature on the three size variation coefficients for the 6 different wood species ( $\bullet$  :  $X = T$ ,  $\square$  :  $X = R$ ,  $\blacktriangle$  :  $X = L$ )

The previous observations are confirmed by Figure 6 where the influence of the pyrolysis temperature on the average relative density variation (of ten samples used for the same test of carbonization) is described. We can therefore affirm that the level of carbonization of a wood sample does not depend on its initial physical characteristics but only on the temperature of pyrolysis. The conversion of the initial oven dry mass of the wood sample into carbon will only depend on the temperature of the carbonization, whatever the wood species is. This observation is in agreement with the literature (Sjöström, 1981) showing that the chemical composition of wood is quite constant and does not significantly vary with the different wood species. The physical properties of carbonized wood are correlated to the initial physical properties

of anhydrous wood: Figure 7 shows that, for all our samples totally carbonized, whatever their initial characteristics are (oven dry density, dimension of the growth rings, presence or not of defects), the density of carbonized wood can always be deduced from the initial oven dry density by the simple relation :

$$\rho_C = 0.63 \rho_{0i} \quad (10)$$

It is also the case for the theoretical porosity (of all samples totally carbonized) which is a linear function of the initial oven dry density (see Figure 8). Whatever the initial wood species and its physical characteristics are, the porosity can be correlated from the simple relation :

$$\varepsilon = 100 - 30 \rho_{0i} \quad (11)$$

Relations (10) and (11) can be useful if charcoal obtained by pyrolysis is used to produce activated carbon. One of the interesting physical properties of activated carbon is its intrasec porosity. Because this porosity can be accurately evaluated by the initial oven dry mass of wood, it is therefore reasonable to determine the ability of some wood wastes to produce activated carbon according to its oven dry density. This last parameter becomes a real criterion of selection for a wood species as far as the structure of carbonized wood is similar to the one of the initial anhydrous wood as shown by Parameswaran and Stamm (1983) and Korytarova and al. (1997).

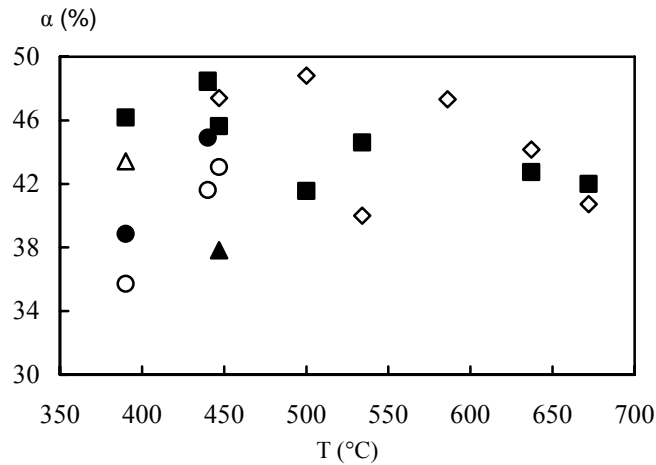


Figure 6. Influence of the pyrolysis temperature on the relative density variation  
 ( $\diamond$  : Ash,  $\circ$  : Douglas,  $\bullet$  : Spruce,  $\blacksquare$  : Poplar,  $\triangle$  : Oak)

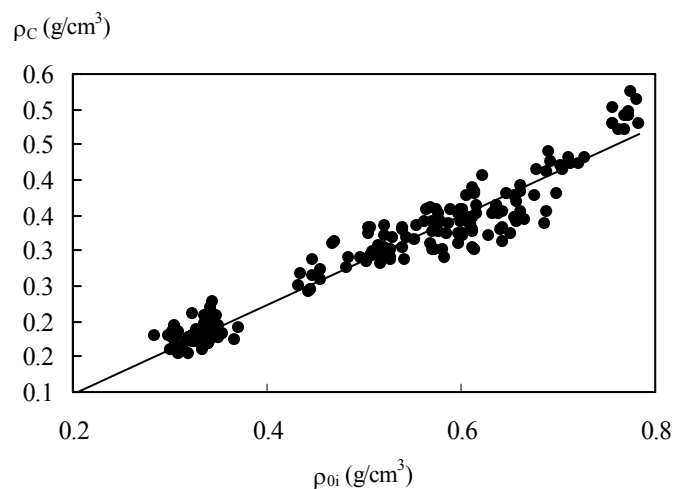


Figure 7. Variation of the density of carbonized wood as a function of the initial oven dry density for all the samples



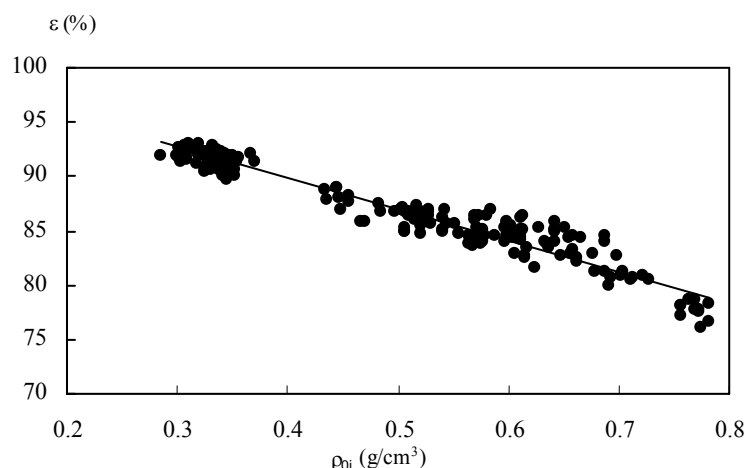


Figure 8. Variation of the theoretical porosity as a function of the initial oven dry density

#### 4. Conclusion and perspectives

Our experimental results obtained by carbonization of 6 different wood species show that, for a constant operating temperature, a pyrolysis gives similar conversion ratio whatever the wood species carbonized is. Even if the initial physical characteristics of the wood samples are very different (initial oven dry mass and thickness of the growth rings in particular), the relative mass loss after a total carbonization is constant and tends to 25 %. It is also the case for the three size variation coefficients. The shrinkage is near 30 % in the tangential direction, 25 % in the radial direction and 20 % in the longitudinal direction. This means that, whatever the wood species concerned is, its conversion ratio into carbon for valorization can be accurately pre-determined by a general law.

Moreover, the initial oven dry density of a wood species, whatever the initial physical characteristics are, can be used as a selection criterion to determine its ability to produce activated carbon. Of course additional tests and measurements must be realized to check the values of the porosity (by mercury porosimetry) and the pore size distribution of carbonized wood. The mass transfer properties of carbonized wood must be also estimated in order to qualify the ability to adsorb some model pollutants.

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