Impact of post-depositional processes on charcoal fragmentation and archaeobotanical implications: Experimental approach combining charcoal analysis and biomechanics

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Impact of post-depositional processes on charcoal fragmentation and archaeobotanical implications: experimental approach combining charcoal analysis and biomechanics

Julia Chrzażvez, Isabelle Théry-Parisot, Gilbert Fiorucci, Jean-Frédéric Terral, Bernard Thibaut

1. Introduction

Charcoal preservation in archaeological sites is variable, even at sites where combustion structures have been identified (Théry-Parisot, 2001, 2010c). According to the current state of knowledge, there is no apparent relationship between charcoal preservation and the sedimentary context. Although this observation does not follow a chronological pattern, it is particularly true for Palaeolithic sites (Braadbaart et al., 2009, 2012; Beresford-Jones et al., 2010; Théry-Parisot et al., 2010a), where identifiable, macroscopic-sized charcoal is at times very rare and/or very altered. In such contexts, charcoal is primarily preserved among the mesoscopic and microscopic unidentifiable fraction (Marquer et al., 2010, 2012), raising the question of the impact of post-depositional processes on charcoal preservation. What are the effects of these processes on anthracological assemblages? Do they have a linear effect on charcoal or are some taxa more fragile than others due to their anatomical structure or chemical composition? This naturally brings us to the question of the representativeness of anthracological assemblages for assessing past vegetation and fuel management.

Since the end of the 1990s, some researchers have focused on a methodical approach to the formation process of anthracological assemblages (Théry-Parisot 1998, 2001; 2013; Théry-Parisot et al. 2010a,b), including (i) human practices, (ii) the physical and chemical modifications of wood during combustion, (iii) depositional and post-depositional processes. Human practices are dependent on non-predictable cultural factors specific to each...
group. On the other hand, combustion and post-depositional pro-
cesses involve physico-chemical and mechanical transformations,
which are independent of socio-cultural contexts, and which affect
archaeological assemblages in terms of mass reduction and frag-
mentation. However, most palaeoenvironmental interpretations
are partly based on variations in the relative frequency of taxa,
calculated by counting the identified fragments in the studied
sample. The effects of combustion on charcoal fragmentation have
been the subject of numerous studies (cf. infra). Conversely, the
effects of post-depositional processes on archaeological assem-
blages are not so well documented. However, all processes, from
trampling to combustion residue, displacement by humans,
weathering, water run off transport, bio or cryoturbation phe-
nomena, alternating freeze-thaw cycles or sediment soaking-
desiccation, can induce charcoal fragmentation (for a better over-
view of sites formation processes see for example Goldberg and
Macphail, 2013). In this paper, we address the effects of deposi-
tional and post-depositional processes and the resulting potential
modification of the assemblages.

The aim of this article is to characterize the physical properties
of several common temperate and Mediterranean European species
in the laboratory, using appropriate measurements on present-day
material. By assessing the differential reaction of each species to
mechanical post-depositional alteration processes, we can evaluate
the palaeoecological representativeness of the archaeological as-
semblages. This study involves 302 samples issued from 10 taxa,
charred at 3 different temperatures and subjected to standardized
compression tests. The results should allow us to assess (i) the
question of the mechanical properties of charcoal, (ii) the frag-
mentation level/rate of charcoal from different taxa and (iii) the
modalities of this fragmentation. The main objective of this study is
to produce data exposing the intrinsic mechanics of the tested
species, to evaluate their conservation potential and to appraise the
possibility of the under or over-representation of certain species in
archaeological contexts.

2. Research history in charcoal fragmentation

After some pioneering studies (Salisbury and Jane, 1940; Santa,
1961; Vernet, 1973; Thébault, 1980), methodological reflections on
the representativeness of anthracological assemblages began in the
approach consisted in analyzing the global fragmentation rate and
the size of the fragments of the different taxa in archaeological
samples. The analysis of Protohistoric archaeological levels (Le
Marduel, Lattes, south of France) yielded fragmentation histograms
with a Poisson distribution for all the taxa within the same layer.
These studies led to the formulation of the “single fragmentation
law” hypothesis for all species (Chabal, 1991, 1997). “A posteriori”
analysis included the undifferentiated fragmentation stages: com-
bustion, post-depositional processes, sampling and sieving of the
material. At the same time, several studies concentrated on the
effects of combustion on anatomy and fragmentation (Rosen and
Olson, 1985; Smart and Hoffman, 1988; Scott and Jones 1991;
Prior and Gasson, 1993; Loreau, 1994; Vaughan and Nichols 1995;
Belcher et al. 2005; Lingens et al. 2005; Braadbaart and Poole,
2008; Théry-Parisot and Chabel, 2010). These works demon-
strated “the non-linearity of species behaviour towards fire sug-
gestings that combustion is a taphonomic agent, which randomly
affects deposits and whose effect on the assemblage is almost
impossible to control” (Théry-Parisot et al., 2010a,b). The impact of
post-depositional processes on anthracological assemblages is a
less-developed aspect of research. Bio-turbation and transport
were mainly studied in the domain of pedo-anthracology (Thinon,
1992; Vaughan and Nichols, 1995; Carcaillet and Talon, 1996;
Blackford, 2000; Nichols et al., 2000; Scott et al., 2000; Scott,
2010; Carcaillet, 2001). Recent work on the impact of pH and
diagenesis on the anthracological material has brought to light a
structural alternation of charcoal in alkaline environments (Schieg
et al., 1996; Cohen-Ofri et al., 2006; Rebollo et al., 2008; Braad-
baart et al., 2009; Ascough et al., 2010, 2011a,b), but also the strong
influence of physical processes on the deterioration of the material
(Braadbaart et al., 2009). Among these physical processes, me-
chanical actions, which directly influence the fragmentation of
charcoal, play a preponderant role. Freeze/thaw laboratory ex-
periments, coupled with measuring resistance to compression, have
shown that wood alteration ante combustion has a strong inci-
dence on the mechanical resistance of charcoal (Théry-Parisot,
1998, 2001). More recently, a study of the mechanical properties
of species from the north of India showed that resistance to
compression and the dimensions of the ensuing fragments is
correlated to charring temperatures (Lancelotti et al., 2010). Me-
chanical resistance to compression is harder when compression is
applied lengthwise to the cross-section and dense wood is more
brittle than less compact woods. In temperate regions, abundant
data are available concerning the properties of wood used for
construction (Ashby, 2005; Forest Products Laboratory, 2010), but
nothing indicates that these data can be transposed to charcoal. For
this reason we developed an experimental study of the mechanical
behaviour of present-day charcoal to evaluate the fragmentation
process of the main species identified in temperate European
anthracological assemblages.

3. Materials and methods

The mechanical tests were carried out in the École des Mines de
Paris (ParisTech CEMEF- Sophia-Antipolis, France). The aim of these
tests was to measure the mechanical response of charcoal to
compression and the fragmentation modalities (number and size of
fragments). The experimental procedure is based on previous work
by Théry-Parisot (1998, 2001)

3.1. Sample preparation

Sample preparation must take account of both the (i) constraints
of the shape of the samples used for the mechanical tests and (ii)
the effect of the physico-chemical alteration on the mechanical
properties of the material during combustion. It generally involves
some form of standardization far removed from the reality of the
studied archaeological contexts. The samples have to present two
perfectly cut parallel sides with no structural, fissure type altera-
tion.

The production of 2 cm cubes, with no charring fissures, is one of
the restrictions of our protocol.

The tests concerned charred samples from 10 taxa commonly
found in archaeological contexts in southern Europe: Acer pseu-
doplatanus, Betula pubescens, Carpinus betulus, Corylus avellana,
Fagus sylvatica, Fraxinus excelsior, Populus tremula, Pinus pinaster,
Pinus sylvestris and Quercus pubescens. The dry wood (12% moisture
content) is issued from branches with a 10–15 cm section.

3.2. Charring protocol

In order to limit sample deformation during charring, each cube
was wrapped in aluminium foil, placed in a porcelain crucible,
covered with sand, and then charred in a muffle furnace. The
charring temperature has a direct incidence on the mechanical
resistance of charcoal (Hillis, 1984; Bengel and Wegener, 1989;
Yildiz et al., 2006; Borrega and Kärenlampi, 2008; Gündüz et al.,
2008; Mburu et al., 2008; Korkut et al., 2008; Korkut and
Hiziroglu, 2009; Kocaefe et al., 2010; Majano-Majano et al., 2012; Poletto et al., 2012). Consequently, the choice of temperature must take into consideration the different stages of the thermal deterioration of the wood (Byrne and Nagle, 1997; Roussel et al., 2006; Braadbaart and Poole, 2008), but must also reflect the probable temperatures of archaeological hearths (Costamagno et al., 2010; Théry-Parisot and Chabal, 2010). The samples were charred at three different temperatures: 400, 500 and 750 °C, in porcelain crucibles placed in the hot oven during 30 min. 302 of the 400 prepared samples were usable, representing a 25% loss principally for samples charred at 400° and 750 °C. On average, 30 samples were tested for each species, with an average of 9.1, 15.6 and 5.5 samples at temperatures of 400 °C, 500 °C and 750 °C respectively.

3.3. Physical characterization

The physical properties of each sample were recorded in order to evaluate their incidence on the fragmentation process. The mass (M for dry wood and MC for charcoal), and the dimensions of the cube were measured in 3 directions, first on air-dried wood and then on charcoal. This allowed for the calculation of the volume (V for dry wood and VC for charcoal), the density using the formula

\[ D = \frac{M}{V} \]  

(WD for dry wood, WCD for charcoal), mass loss (M – MC)/M (Mloss %), volume loss (V – VC)/V (Vloss %) and density loss (WD – WCD)/WD (Dloss %).

Systematic porosity measurements were taken on the transverse sections of charcoal samples using SEM image analysis. The porosity ratio corresponds to the difference in porosity between the initial and final wood. These measurements were incorporated into the multivariate analysis to include the impact of the porosity of taxa on their mechanical properties and fragmentation. A macro for this measurement was written with image J software (Ducom, 2010).

3.4. Compression procedure

A hydraulic traction-compression testing machine, equipped with a 10 kN sensor (Instron 1121), was used for the compression experiments. Pressure was applied parallel to the fibers. The descent speed was fixed at 0.1 mm/s during 30 s from the time of contact with the sample, which was placed on its transversal side. The pressure applied and the resulting decrease in the height of the sample (displacement of the tray), were recorded during the tests. These values allowed us to plot the stress (MPa)/strain (%) curves, by dividing the force by the surface of the transversal side of the sample (stress), on one hand, and the displacement by the height of the sample (strain), on the other hand.

Fig. 1 represents a typical test curve. It is comprised of three main parts:

- a gradual rise after the initial contact, which corresponds to setting up the compression;
- a rapid and linear rise of the stress until the peak, which corresponds to the crushing of the whole sample;
- a more or less brutal decrease in stress, possibly with non-negligible drag strain, corresponding to the destruction of the charcoal structure.

It is noteworthy that compression is never reversible; it is not therefore an elastic phenomenon.

This diagram allows us to calculate several mechanical indicators in phase 2:

- Maximum stress called crushing strength (CS in MPa);
- Crushing modulus (CM in MPa);
- Theoretical crushing strain (TS = CS/CM).

Crushing modulus (CM), like any modulus, relates charcoal contraction to the compressive force exerted on it. If CM is low, the charcoal is less rigid during compression.

As both wood and charcoal are honeycomb-like materials, mechanical properties in grain direction are roughly proportional to their density (Gibson and Ashby, 1999). It is therefore interesting to use “specific” crushing strength, i.e. CS divided by charcoal density: CS/WCD (expressed in 10^3 m^2/s^2), as a criterion, independently of density itself.

The applied pressure induces sample fragmentation. The residues of each test were systematically sieved in three different meshes: [1 – 2 mm], [2 – 4 mm], > 4 mm and the fragments were then counted using image analysis (ImageJ).

3.5. Statistical methods

Several univariate to multivariate statistical methods were used to analyze the data and to assess the relation between the mechanical properties and the fragmentation process. The choice of tests depends on the purpose of the analysis and the nature of the available data. Non-parametric tests based on the ranks of the values were chosen when the normality of the distribution was not verified. The Spearman’s rank correlation coefficient is a nonparametric measure of statistical dependence between two variables. Regression analysis was used when the dependent variable follows a Normal distribution. The very flexible and extremely powerful one-way analysis of variance (ANOVA), widely used for the analysis of experimental schemes, can simultaneously analyse several scenarios and detect the effects of independent nominal variables on a large number of continuous variables. This test was performed to assess and evaluate the effect of the charring temperature and the taxon on the fragmentation process. The Bonferroni–Dunn test allows for comparisons, controlling the family error rate. It was carried out in order to identify groups of taxon with the same fragmentation process. A multiple correspondence analysis (MCA) was applied to our data set categories to test relationships between variables.
4. Results (Table 1)

4.1. Physical and mechanical properties

4.1.1. Mass, volume and density

In our tests, charring induces severe mass, volume and density loss.

If we disregard wood water loss at the beginning of charring (on average 12%), on average, mass loss (Mloss) approaches 55% at 400°C, 60% at 500°C and 70% at 750°C. There are no systematic significant differences between the different species.

Volume loss (Vloss) is about 50% at 400 and 500°C and increases to 58% at 750°C, which is considerable in comparison to the hygroscopic shrinkage between air-dried wood and oven-dried wood (about 6–8%). In this case, there are noticeable systematic differences between species, with Quercus pubescens displaying the lowest volume shrinkage and Carpinus betulus the highest.

Density loss (Dloss) increases markedly with temperature (Fig. 2a). It rises from 32% at 400°C, to 40% at 500°C, and reaches 55% at 750°C. Average density loss is 40%, with variations from 33 to 50% according to the species (Fig. 2b), with Quercus and Carpinus representing the two extremes (Quercus records a much higher density loss than the other species — which tends to slot it into the average — whereas that of Carpinus is much lower).

Species T°C Nb WD WCD Mloss Vloss Dloss >4 mm 2–4 mm 1–2 mm Total CM CS TS CS/WCD Means

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</table>

Table 1

Average values for mechanical properties and fragmentation of the 10 species and 3 heat treatments. Abbreviations: Nb: number of samples; WD: wood density; WCD: charcoal density; Mloss: mass loss; Vloss: volume loss; Dloss: density loss; >4 mm, 2–4 mm, 1–2 mm: number of fragments in each class size; Total: total number of fragments; CM: crushing modulus; CS: crushing strength; TS: theoretical crushing strain; CS/WCD: specific crushing modulus.

4.1.2. Crushing strength (CS), crushing modulus (CM) and theoretical crushing strain (TS) (Fig. 3)

■ The effect of temperature

The three mechanical indicators evolve markedly with temperature (Table 1). All three decrease when the charring temperature increases, with crushing strength (CS) being more affected than the others. The crushing modulus (CM) decreases slightly between 400°C (424 MPa) and 500°C (396 MPa), but is almost halved at 750°C (226 MPa) (Fig. 3a). Crushing strength (CS) varies on average from 16.9 MPa at 400°C, to 14.5 MPa at 500°C and 6.9 MPa at 750°C (Fig. 3b). As for the theoretical crushing strain (TS), it decreases regularly from 4.3% at 400°C to 3.2% at 750°C, with a value of 3.8% at 500°C (Fig. 3c).

It appears from these results that charcoal becomes less resistant and more fragile as charring temperatures increase.

Charring tends to regulate density differences between species but charcoal density remains globally proportional to the initial wood density (Fig. 2c). The Spearman correlation test indicates a significant positive correlation between the two series of values (rho = 0.69/p-value < 2.2e-16) (Table 2).
Species effect

Due to the fact that a larger and more constant number of specimens were charred at 500 °C, the mean values at this temperature were used to compare species.

The three indicators vary according to the different species (Table 1), which partly explains the substantial standard deviations observed for this temperature. The differences between taxa are noticeable, with CM ranging between 285 MPa for Quercus and 591 MPa for Carpinus, CS between 7.8 MPa for Quercus and 20.3 MPa for Corylus and TS between 2.8% for Quercus and 4.7% for Corylus. Quercus is by far the least resistant and most fragile taxon, while Acer, Carpinus and Corylus are the most resistant and the least fragile. The difference is even more accentuated between Quercus and Corylus for the specific crushing strength: respectively 17.1 and 51.3.

Interaction between the effects of temperature and species

An increase in temperature globally induces a decrease in charcoal resistance (CS), regardless of species (Fig. 3). This decrease is partly due to the reduction in charcoal density, which is most pronounced between 500 and 750 °C.

According to the CIRAD database (Cirad database TROPIX® 7, 2011), measurements taken on 243 air-dried wood samples from tree species, with densities ranging from 0.2 to 1.2, show that resistance to axial compression is proportional to the density of dry wood (WD): $CS = 84.7 \times WD$, with a coefficient of regression $R^2 = 0.88$. The CS/WD ratio is called specific resistance (in relation to density) and varies little between species.

It is therefore interesting to compare the specific resistance of charcoal (CS/WCD) to the mean value observed for air-dried wood. On average (Table 1), the ratio between (CS/WCD) and this mean value (84.7) is still 0.48 and 0.44 for 400 °C and 500 °C respectively, but it drops to 0.28 at 750 °C.

There are marked differences in temperature between species when the sudden drop in resistance occurs (Fig. 4). Fraxinus, Fagus and Quercus already display low values at 400 °C and these values do not fall suddenly at 750 °C. Pinus pinaster decreases clearly between 400 and 500 °C but not so much between 500 and 750 °C. All the other species present a strong decrease in resistance between 500 and 750 °C.

4.2. Fragmentation

4.2.1. Global fragmentation process, all class sizes combined

After the compression tests, the samples produced on average, 170 fragments at 400 °C, 200 fragments at 500 °C and 165 fragments at 750 °C (Fig. 5a). The relationship between temperature
and the number of fragments is therefore not consistent during our tests. The total number of fragments for each species presents marked variability, ranging from 34 fragments for *Populus* to 400 for *Fraxinus*, with, on average, 101 fragments for *Populus* and 246 for *Quercus pubescens*.

The differences between species are significant, as shown by the results of the ANOVA (Table 3), which make it possible to classify the effects of temperature and species. Both factors combined account for 61.5% of the total variability but the $F$-value and the sum of the squares for the SPECIES factor are more significant, explaining 49.7% ($R^2$) of the variation of the model. The histogram (Fig. 6) summarizing the total number of fragments produced by compression, according to species and temperature, clearly shows the preponderant role of the taxon on fragmentation. Apart from some rare exceptions (500°C/*Fraxinus*), the observed variability is much more significant between the different species than between temperatures.

A bilateral paired-comparison test brings to light four partly overlapping groups of taxa. *Populus* on one hand, and *Quercus* and *Fraxinus* on the other hand, display contrasting responses, which differ from those of most species. On average, *Populus* is not very fragmented whereas *Quercus* and *Fraxinus* are much more fragmented. The other species are clustered around two median groups. *Fagus* is similar to *Populus*, followed by a group made up of *P. pinaster, Acer* and *Betula*. *P. sylvestris, Corylus* and *Carpinus* tend to display slightly greater fragmentation and *P. sylvestris* has median fragmentation (Table 4).

### 4.2.2. Mechanical properties versus fragmentation

Series of Spearman tests show that, on the whole, there is virtually no link between mechanical properties and the fragmentation process (Table 2). Mass loss, crushing strength and specific crushing...
strength are not correlated with the total number of fragments produced. The crushing modulus is very slightly correlated with the total number of fragments: the p-value is significant (0.0076), but the determination coefficient is low (\(\rho = 0.15\)). Scatter graph (Fig. 7) explicitly shows that there is no relation between crushing strength (which is a good expression of the brittleness of charcoal) and the number of fragments produced by compression tests. In other words, mechanical properties and fragmentation are strongly dependent on the species, but there is no correlation between the two factors themselves. Each species is more or less resistant to compression but once the fragmentation process has begun, it affects each species independently of the pressure applied.

4.2.3. Fragmentation modalities by class size [1–2 mm], [2–4 mm], >4 mm

The first observation concerns the disparity in fragment distribution between the different class sizes. All species combined, the average number of fragments by class size is 111 in the smallest class size [1–2 mm], 51 in the second [2–4 mm], and only 19 fragments larger than 4 mm (Fig. 5b).

According to the species, the number of fragments varies from 16 to 304 in the class [1–2 mm], from 11 to 121 in the class [2–4 mm] and from 5 to 62 in the class >4 mm (Fig. 5c). The variance of the three series is thus quite marked and the coefficient of variation is around 40% for the three class sizes.

Fragmentation variability by species is very significant in the class [1–2 mm] with substantial tiering of the dispersion boxes (Fig. 5a). Populus is the least fragmented species with a minimum of 16 fragments and Fraxinus is the most fragmented species with a maximum of 304 fragments. The bilateral paired-comparison test (Bonferroni–Dunn procedure, Table 4) distinguishes four significantly different groups.

The upper and lower extremes are respectively made up of an isolated species, Populus, and a group made up of Corylus, Quercus, Carpinus, Fraxinus, which generally yields more fragments. Fagus and P. pinaster are similar to Populus, whereas Acer, P. sylvestris and Betula display intermediate fragmentation, between the species with high and low fragmentation. Variability is less marked in the intermediate class [2–4 mm], where divergence between the species is slightly reduced and only two species deviate: Populus generally produces less fragments (mean = 33) and Quercus (mean = 78) produces on average a lot more fragments. As for the other species, Betula, Fagus, Carpinus and Corylus present a low fragmentation tendency whereas Fraxinus, P. Sylvesteris, P. pinaster and Acer display a high tendency (Fig. 8b).

Lastly, in the >4 mm class, the dispersion boxes have a more or less constant distribution, apart from Quercus, which is quite detached from the other species (Bonferroni–Dunn-test). Variability is generally much less significant here (Fig. 8c).
Differences between taxa are thus very slight in the >4 mm size and relatively minor in the [2–4 mm] class. They are most marked in the [1–2 mm] class.

### 4.3. Multivariate analysis

The multiple correspondence analysis (MCA) conducted on all the specimens charred at 500 °C allows us to discuss the relationships between our results and the main physical and anatomical characteristics of the studied species. Only the variables with a strong impact on the analysis have been retained (defined by two or three modalities: high/medium/low). For example, fiber thickness was initially tested and then removed. Moreover, the species themselves are integrated as supplementary variables and are not used for the calculation of the ACM (Fig. 9).

On axis 1, accounting for 62.21% of the total variance, the “large vessels”, “very large and long rays” and “high porosity” modalities are closely linked. These modalities are secondarily associated with the absence of spiral thickenings and are related to a low crushing strength (CS) and a high number of fragments >4 mm. On axis 2, in the upper right part of the graphic, in relation to the active variables on axis 1, “high density” is strongly correlated with the high values of the total number of fragments and the high number of fragments >1 mm.

4.4. Results synthesis

Table 3 summarizes correlation coefficients and brings to light the differential responses of the tested species as regards resistance to compression and fragmentation. These two processes are clearly not related. We have observed that:

- Temperature increase generally induces a decrease in the resistance of the charcoal, whatever the species (Fig. 3). This is similar to the results of tests carried out at low temperatures on industrial wood (Gündüz et al., 2008; Korkut et al., 2008; Korkut and Hiziroglu, 2009; Kocaefe et al., 2010; Majano-Majano et al., 2012). Crushing strength loss is particularly noticeable above 500 °C.
- Density loss is correlated to the charring temperature but is also related to the species. However, charring tends to attenuate the differences in density between species (Fig. 2).
- Density loss induces crushing strength loss and accounts for the main mechanical properties of charcoal.
- All temperatures combined, charcoal resistance is also linked to the species. The mechanical properties of the species are modified at distinctive thermal thresholds (Fig. 3).
- Resistance to pressure is dependent on the species but fragmentation intensity is not correlated to the resistance of the material itself. The least resistant species are not those subject to the most fragmentation (Fig. 7).
- The relationship between the number of fragments produced by compression and temperature is not constant (Fig. 5a).
- The total number of fragments after compression depends mainly on the species, regardless of temperature (Table 3 and Fig. 6).

### Table 3

Effect of species and temperature on fragmentation (ANOVA one-way variance analysis and PLSD Fisher test).

<table>
<thead>
<tr>
<th>Variable</th>
<th>DDL</th>
<th>Sum of the squares</th>
<th>F value</th>
<th>Pr &gt; F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Species</td>
<td>9</td>
<td>671323.8</td>
<td>40.762</td>
<td>&lt;0.0001</td>
</tr>
<tr>
<td>Temperature</td>
<td>2</td>
<td>45851.4</td>
<td>25.054</td>
<td>&lt;0.0001</td>
</tr>
</tbody>
</table>

### Table 4

Groups of species vs. fragmentation (Dunn–Bonferroni Test).

<table>
<thead>
<tr>
<th>Total</th>
<th>[1–2 mm]</th>
<th>[2–4 mm]</th>
<th>&gt;4 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Taxon</td>
<td>Groups</td>
<td>Groups</td>
<td>Groups</td>
</tr>
<tr>
<td>Populus</td>
<td>A</td>
<td>Populus</td>
<td>A</td>
</tr>
<tr>
<td>Fagus</td>
<td>B</td>
<td>Fagus</td>
<td>A</td>
</tr>
<tr>
<td>P. pinaster</td>
<td>C</td>
<td>P. syl.</td>
<td>A</td>
</tr>
<tr>
<td>Betula</td>
<td>B</td>
<td>Acer</td>
<td>B</td>
</tr>
<tr>
<td>Acer</td>
<td>C</td>
<td>P. pinaster</td>
<td>C</td>
</tr>
<tr>
<td>P. syl.</td>
<td>C</td>
<td>Betula</td>
<td>C</td>
</tr>
<tr>
<td>Corylus</td>
<td>C</td>
<td>Corylus</td>
<td>D</td>
</tr>
<tr>
<td>Carpinus</td>
<td>D</td>
<td>Quercus</td>
<td>D</td>
</tr>
<tr>
<td>Fraxinus</td>
<td>E</td>
<td>Carpinus</td>
<td>D</td>
</tr>
<tr>
<td>Quercus</td>
<td>E</td>
<td>Fraxinus</td>
<td>D</td>
</tr>
</tbody>
</table>

In the upper left part, the high crushing strength is strongly correlated to the presence of spiral thickenings and, secondarily, to the “small aggregate vessels” modality.

Lastly, in the lower part of the graphic, the “low density” (WCD) and “small isolated diffuse vessels or homoxylous wood” are linked to the low total number of fragments.

Wood with a marked porous zone, with large vessels, such as Quercus and Fraxinus, and with long radial file vessels (Carpinus and Corylus) are the most fragmented. Homogeneous wood (small isolated diffuse vessels or homoxylous) such as Populus, Acer, Betula or P. pinaster, P. sylvestris are those that produce fewer fragments. Corylus, Carpinus and Acer present a higher crushing strength than the other taxa. This characteristic seems to be linked to the presence of spiral thickenings. Conversely, the weak value of crushing strength is either linked to the porosity of the charcoal (case of Fraxinus and Quercus), or to its low density, as is the case for Populus, P. sylvestris and P. pinaster. In all cases, wood with a low density presents a low value for crushing strength. On the other hand, dense wood alternatively yields either very high or very low values.
Fig. 6. *Populus* is not prone to much fragmentation whereas *Quercus* and *Fraxinus* produce a lot of fragments. Between these two extremes, *Fagus*, *P. pinaster*, *Acer* and *Betula* display little fragmentation, whereas *Corylus* and *Carpinus* tend to be highly fragmented. *P. sylvestris* shows an intermediate response to fragmentation (Table 4 and Fig. 5c).

- However, interspecific variability is significant for the small fragments [1–2 mm], but less so for larger fragments [2–4 mm]. In the largest class size (>4 mm), only *Quercus* is different from the other taxa, producing a lot more fragments (Fig. 8).
- Wood with a marked porous zone, with large vessels, and with long radial file vessels (*Carpinus* and *Corylus*) produces the most fragments. Homogeneous wood (small isolated diffuse vessels or homoxylates) such as *Populus*, *Acer*, *Betula* or *P. pinaster*, *P. sylvestris* are those that produce the least fragments (Fig. 9).
- The presence of spiral thickenings (*Corylus*, *Carpinus* and *Acer*) is linked to a higher crushing strength. The low value of crushing strength is either linked to the porosity of the charcoal (*Fraxinus* and *Quercus*), or to its low density (*Populus*, *P. sylvestris* and *P. pinaster*). In all cases, wood with a low density presents a low value for crushing strength. On the other hand, dense wood alternatively yields either very high or very low values (Fig. 9).

5. Interpretation

5.1. Focus on factors impacting fragmentation

- Density

The density of dry wood seems to have an impact on fragmentation: charcoal from dense wood is very fragmented while low to medium density wood charcoal presents low to medium fragmentation. Despite a low coefficient of determination (0.267), there is a significant positive correlation (Rhô = 0.517) between “wood density” (and therefore charcoal density) and the “total number of fragments”. As the majority of the fragments are in the [1–2 mm] fraction, density is also logically correlated with the number of small fragments, as shown by the MCA analysis: the denser the wood, the higher the number of small fragments.

- Wood porosity vs. homogeneity

Fragmentation intensity is related to charcoal porosity. Cellular organization seems to be strongly linked to the fragmentation processes. Marked porous zones with large vessels or radial pore files are prone to fragmentation. Conversely, more homogeneous porous wood (with small isolated diffuse vessels or homoxylates) such as *Populus*, *Acer*, *Betula* or *P. pinaster*, *P. sylvestris* tends to be less fragmented.

- Length and width of the rays

The production of large fragments, as for *Quercus*, is also linked to the presence of a significant porous zone. But *Quercus* differs from the other species by the presence of multiseriate rays, which create fragile zones after combustion and account for the specific fragmentation mode of this species. For the other species, the rays, whether multiseriate or uniseriate, play a secondary role in both fragmentation and the mechanical properties (cf. central position and weak contribution in the construction of the MCA graphic).

5.2. Factors affecting crushing strength

In all cases, charcoal with a low density presents a low value for crushing strength, but high density charcoal alternatively yields...
very high or very low values. The relationship between charcoal density and crushing strength is thus not clear.

*Corylus*, *Carpinus* and *Acer* present a higher crushing strength than the other taxa. This characteristic is linked to the presence of spiral thickenings, which are preserved after charring. Conversely, the weak value of crushing strength is either linked to the porosity of the charcoal, in the case of *Fraxinus* and *Quercus*, or to its low density, as is the case for *Populus*, *P. sylvestris* and *P. pinaster*.

For dry wood, crushing strength is more or less proportional to wood density. Although *Quercus* is the wood with the highest density, its charcoal has the lowest crushing strength. This is due to the fact that charring induces the highest density loss for this species, perhaps because of its chemical composition. In addition, *Quercus* displays two orthogonal arrays of weak zones: an initial porous zone in a tangential direction and very large rays in the radial direction. This should be conducive to the initiation of a large number of cracks at rather low stress levels.

On the other hand, *Corylus*, *Carpinus* and *Acer* present a higher crushing strength than the other taxa, although the density of these species is no higher than that of *Fraxinus*, for example. They are more homogeneous woods with less distinct weak zones prone to fractures. Furthermore, the presence of spiral thickenings in the fibers, which are preserved after charring, appears to reinforce the structure of these charred woods.

*Populus* is characterized by a very homogeneous structure, but with a much lower density than the former species.

The low resistance of *Fraxinus* and *Fagus* charcoal, in spite of the rather high density of these woods, can also be explained by the presence of weak zones: tangential (initial porous zone) for *Fraxinus* and radial (large rays) for *Fagus*.

6. Discussion: from experimentation to the archaeological context

This study shows that the anatomical structure of the different tested wood taxa has a significant impact on the mechanical properties of charcoal. Charring produces less heterogeneous material than the different initial woods, but it does not erase the differences in density, organization and cellular composition, etc.

Thermal response, resistance to pressure, fragmentation and its modalities are largely dependent on the anatomical features and thus, on the physical characteristics of wood. Some of the noted differences are significant from an archaeological viewpoint. The alteration of the mechanical properties follows a differential thermal pattern depending on the species, which can cause differences in taxa fragmentation in the same hearth or among scattered charcoals. The formation temperature of charcoal also influences the fragmentation of the material (Théry-Parisot, 2013). Yet, the temperatures are highly fluctuant in a same combustion structure. It is therefore very difficult to archaeologically assess the question of the weakness of charcoal in relation to charring temperatures.

Fragmentation differences are mainly visible outside the [>4 mm] class size. These first results tend to show that anthracological analysis limited to charcoal over 4 mm would induce less risk of under or over-representation of the different taxa, with the exception of *Quercus*, which is over-represented in this class size.

We also noted variations in crushing strength values from one species to another. This signifies that the rupture of the material is not initiated at the same pressure levels. Thus, from an archaeological perspective, in the same burial conditions, taxa will not necessarily demonstrate the same resistance capacity, which could

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Fig. 8. Box & Whiskers plot for the number of fragments by species in the three class sizes: 1–2 mm (a); 2–4 mm (b); >4 mm (c).
bias interpretation. However, crushing strength depends on the formation temperature of charcoal and has no impact on the quantity of fragments produced after the rupture of the material. This results in a set of extremely variable situations where it is impossible to assess or to measure all the parameters. It is thus important to study and to take account of burial conditions (the nature and intensity of post-depositional processes involved in the deposit formation, rate of burial at the scale of the site, but also intra-site variations), but it is nonetheless impossible to infer the mechanical behaviour of charcoal. However, when interpreting the data, it is imperative to bear in mind that some taxa are more fragile than others.

Conversely, the values obtained for crushing strength appear to be more significant. Charcoal tolerates relatively high pressure before fragmentation, oscillating between 8 and 19.4 MPa, with an average of 13.7 MPa. Mechanically, charcoal is considered to be fragile. In comparison, the crushing strength of common conifers (uncharred) is between 18 and 30 MPa, that of ordinary concrete is between 16 and 40 MPa and that of steel between 235 and 350 MPa (Ashby, 2005). Yet most post-depositional processes do not generate very strong pressure. Cattle exert greater static pressure (160–192 kPa) on soil than sheep (83 kPa), although this pressure is at least doubled when animals are walking (Drewry, 2006), which represents less than 0.4 MPa. The value for human beings cannot be much higher. The maximum pressure measured during freeze propagation experiments in a limestone notch is about 6 MPa (Bost, 2008).

Thus, the intense fragmentation of charcoal (extending at times to the disappearance of the macroscopic fraction), characteristic of some Pleistocene sites (Théry-Parisot, 2001; Théry-Parisot et al., 2010a; Beresford-Jones et al., 2010, Marquer et al., 2012) does not seem to result from single, short-term processes. However, classic processes, such as freeze-thaw action or trampling occurring repeatedly during the course of a phase of low (or no) sedimentation, could doubtlessly generate progressive charcoal weakening, leading to extreme fragmentation. Under natural conditions, the repetition of these processes induces progressive mechanical fatigue of the material (which is not measured in our experiment), thereby increasing charcoal damage. Finally, it is important to distinguish post-depositional processes from fuel management. The disappearance of the coarse fraction due to burial conditions sometimes leads to the misinterpretation of charcoal scarcity, suggesting that other fuels, such as bones or dung, were used. Further investigations based on the study of very thin coarse fractions combined with geoarchaeological studies, are required, so that more reliable interpretations can be proposed. (Fernández-jalvo et al., 2010; Marquer et al., 2010, 2012; Miller et al., 2010; Scott and Damblon, 2010).

Naturally, these observations must be moderated, notably because of the way the samples are made, which is quite far removed from the archaeological reality. Complementary analyses are required, incorporating other criteria, such as the chemical composition of the taxa. Moreover, this paper does not take account of the other stages of fragmentation, namely combustion. Through cross indexing data from research on combustion and post-depositional processes, it will become possible to gain a better understanding of anthracological assemblages.

Based on an innovative experimental approach combining both archaeobotany and biomechanics, these results are fundamental for a comprehensive understanding of archaeology, archaeobotany and palaeoecology. They document the mechanical behaviour of charcoal, a largely unknown domain up until now. They enhance our understanding of charcoal taphonomy by providing added resolution and improving the accuracy of charcoal analysis.