

Influence of different parameters in the fire behaviour of seven hardwood species

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Abstract

Wood is a widely used material in the construction sector, both in structural and non-structural applications. Tropical species are appreciated for their high quality and durability in furniture, outdoor and indoor claddings and floors. However, limited information exists about fire reaction of tropical wood. Density is one of the factors that influence the rate of pyrolysis reactions and consequently the charring rate. However, other wood characteristics such as the mineral content also exert an influence on the pyrolysis combustion of wood. Hardwoods present a complex morphological structure and a significant amount of minerals, extracts and exudates. In this work, we study the fire reaction of seven species of tropical hardwood using various fire tests. Results are compared from one test to other one and are discussed in relation with physicochemical properties of wood species. The results show that although there is some correlation between high density and ignition time, parameters such as morphology and mineral content are also relevant.

Keywords: fire behaviour, hardwood, pyrolysis, flammability, thermal stability.

1. Introduction

The use of wood in buildings, not only in structural applications, is object of increasing interest. In the market, it is possible to find a huge variety of wood species. The use of tropical wood species is increasing in some European countries for the high qualities they can offer, especially for structural and separating elements, interior claddings, flooring and furniture. Several articles treat the fire resistance and charring rate of tropical woods [1, 2], but limited literature can be found about the fire reaction of this kind of wood. In applications such as cladding or furniture fire reaction is an important aspect to evaluate the contribution of wood in case of fire.

Generally, wood density is considered one of the most important factors for achieving a better fire behaviour due to its influence on the rate of pyrolysis reactions and consequently on the charring rate[3].When a wooden piece is exposed to fire, it undergoes thermal degradation, including different and complex thermo-physical and thermo-chemical processes such as heat transfer, drying, pyrolysis, charring, mass loss and smouldering. The morphology and the chemical components of the wood species strongly affect all these processes [4].

Wood is a natural composite with a set of specialized cells that perform important functions as providing mechanical strength to the branches, conducting essential liquids and storage of nutrients reserves. Cellulose, hemicellulose, lignin, extractives and exudates are the main components of wood. Cellulose is a polymer conformed by a high number of glucose units. The degree of polymerization (DP) varies depending on the wood species, but cellulose from hardwoods has usually a DP above 3000 [5]. Hemicellulose is formed by low molecular weight polysaccharides with a DP of around

200[6] and Lignin, the third polymeric constituent of wood, is a cross-linked aromatic polymer[7]. These differences in the molecular composition explain the lower thermal decomposition of hemicellulose, between 180-350°C, followed by cellulose (275-350°C) and lignin (250-500°C) [8]. Several authors have described the different stages occurred during combustion of wood [9][4]:

- 100 - 200°C: release of water and non-combustible gases.
- 200 - 300°C: pyrolysis of hemicellulose and lignin and production of charred wood.
- 300 - 455°C: depolymerisation of cellulose and lignin and release of flammable gases.
- 450°C: oxidation and smouldering of the charred wood residue.

Extractives and exudates exert a significant influence on essential characteristics of trees such as the variety of colour, smell, density, dimensional stability, durability and resistance to attack by fungi and insects[10]. These compounds can also affect the calorific value and, therefore the flammability [11]. Trees also contain different minerals, which make a fundamental contribution in their metabolism. Most minerals are absorbed from soil through its root system, while others are absorbed from air through the leaves. Some of the metallic elements found in the mineral salts are: Al, Ca, Fe, K, Mg, Na, Mn and Ni. These elements are combined to form oxides, carbonates, silicates, chlorides, phosphates, nitrates or sulphates. These minerals can play a significant role in the pyrolysis process of wood through a catalytic effect that influences the temperatures of pyrolysis and the products released [12][13]. Total inorganics in wood can be evaluated by determination of ash content after combustion of the material. Zule et al. indicate that woods typically contain from 0.1 to 1.5 % of ash, but these contents may be higher in some tropical species [14]. Torelli et al. studied 43 Mexican tropical hardwoods and they found amounts of silica up to 4.7% for one of

the species [15]. In the case of Avila-Calderon et al. the evaluation of the ash content on 3 Mexican woods revealed ash contents of 6.2% for one of the studied species [16]. Which indicates that tropical woods are especially rich in different inorganic constituents.

This work provides a detailed study of the fire reaction of seven species of tropical hardwood using various fire tests including cone calorimeter. The species selected have a wide range of densities, as well as diverse physical and chemical characteristics. This allows us to carry out a complete study to observe the influence of different parameters on the fire behaviour **and provide data about these species which is not available yet.**

2. Materials and methods

2.1 Materials

Seven species of tropical hardwoods from Mexico have been included in this study. Table 1 summarizes the seven hardwoods species studied in this research. Even though all the samples are hardwoods, there exist remarkable differences in their densities as well as in their colour and surficial aspect.

Wood samples were conditioned at 21°C and a relative humidity of 55% prior to their characterization. The equilibrium moisture content of the samples is shown in table 1. As it can be observed there are some differences in the equilibrium moisture values being the denser wood species the ones with less moisture content.

2.2 Characterization methods

In order to evaluate the thermal and fire behaviour of the wood samples the tests described below were performed. Scanning electron microscopy was used to evaluate the morphology of the different wood species as well as the presence of mineral elements.

Thermogravimetric analysis

Thermogravimetric analyses (TGA) were performed using a furnace coupled with a precision scale. This device allowed testing larger samples, between 0.3 to 1.5 grams, than the conventional thermogravimetric equipment, which is interesting for heterogeneous samples. The samples were heated at 4°C/min until 1000°C under air atmosphere.

Limiting Oxygen of Index (LOI)

The Limiting Oxygen Index (LOI) was determined following the standard UNE-EN ISO 4589. Although LOI test primarily was developed for plastics, this method has been successfully used to determine fire performance of wood [15]. The standard establishes the test procedure to determine the LOI on vertically oriented small specimens, in this case of 70 mm x 10 mm x 1 mm, under a mixture of nitrogen and oxygen flow. LOI is the minimum concentration of oxygen at which a specimen will support combustion under the criteria defined in the standard.

Epiradiator test

A radiator device described in the Spanish UNE 23725-90 standard was employed to evaluate the time to ignition and the ability to extinguish the flame after the heating source was removed from the sample. Specimens (70 x 70 x 10 mm) were placed on a metallic grid 3cm below a heat source of 500 W, which was taken away and put back after each ignition and extinction. Under this test conditions the heat flux on the surface of the sample is **30 kW/cm²**. Three specimens of each wood species were tested, and the parameters determined were the number of ignitions (No), the time at which the first ignition occurs (t_0) and the average time of flame persistence during the first 5 min of combustion (t_m).

Pyrolysis combustion flow calorimeter (PCFC)

A pyrolysis combustion flow calorimeter from Fire Testing Technology was used to measure the heat release rate as a function of the temperature of the different wood samples. The equipment consists of a pyrolysis chamber where the samples were heated under nitrogen atmosphere up to 750°C at 1°C/s. The evolved gases were then transported by an inert gas to the combustor that works at 900°C in a flow of oxygen (20cm³) and nitrogen (80cm³).

Cone calorimeter

The cone calorimeter tests were carried out following the procedures indicated in the ISO 5660 standard with a FTT cone calorimeter. The testing procedure was adapted to test square specimens of 70 × 70 × 10mm³ with a heat flux of 35kW/m². Each species was tested twice. Good reproducibility was obtained.

Scanning Electron Microscopy (SEM)

The morphology of the samples was studied by scanning electron microscopy using an Environmental microscope, ESEM Quanta 200 FEI. Elemental analysis using energy dispersive X-ray spectroscopy (EDS) was also performed to identify the elements present in the mineral crystals observed inside the wood cells.

In order to obtain more information about the amount and nature of the inorganic phases, the different wood samples were calcined at 800°C during 1h to obtain the residue. This residue was also observed and analyzed by SEM and EDS respectively.

3. Results and discussions

Since the density of the wood species ranges from 448 to 1234 kg/m³, it is expected that this parameter will influence greatly the fire behaviour. Nevertheless, the internal

structure differs significantly from one wood species to the other one. The morphology of the different wood samples was examined with SEM (Figure 1). It was observed that LA, CA and TC have a more compact morphology compared with the other samples. This is not surprising in woods species with high density as CA and TC; however, it is remarkable in a medium density wood as LA. Tangential section of LA shows mostly uniseriate and biseriate rays and seldom triseriate rays. Many cells in uniseriate rays are saturated with minerals and also some saturated tracheids can be found. This saturation may influence the oxygen and gases flow during the combustion.

Wood samples were calcined in order to concentrate and analyse the minerals present in each wood species (Figure 2). Table 2 shows the amount of solid residue and the nature of main elements contained in each sample after calcination and detected by EDS. From the crystal habit it can be deduced that samples EC, SH, CA contain whewellite, a monohydrated calcium oxalate. This is in good agreement with the formation of calcium oxide in the calcination of the wood, which is subsequently transformed to calcium hydroxide or calcium carbonate. In sample LA, the presence of two types of inorganic compounds was also observed. One of the substances was rich in sulphur and potassium, probably a form of potassium sulphate, and the other one contained calcium as the main element. Several authors reported the influence of the inorganic compounds in the modification of the thermal decomposition and pyrolysis of lignocellulosic products. T. Hosoya et al. mentioned the influence of inorganic substances on the higher production of glycoaldehyde, hydroxyacetone and carbonized products during the wood pyrolysis [16]. K. Raveendran et al. found that the amount of potassium or zinc together with the lignin content modify the pyrolysis of biomass [17]. In general, inorganic matter is believed to increase char formation and inhibit the formation of volatile

products [18]. On the other hand, S. Liidakis et al. did not detect a significant influence of the inorganic phases on the ignitability of wood [19].

The thermogravimetric curves of the different wood samples are shown in Figure 3. The first weight loss step ($< 150^{\circ}\text{C}$) is assigned to the release of water. The degradation after 250°C occurs in several steps for all species. In this range of temperatures ($250\text{-}400^{\circ}\text{C}$), there is a significant relation between the thermal stability and the density. The less dense species, TD, decomposes at significantly lower temperatures than the rest of the wood samples. This is probably due to the presence of high levels of hemicellulose and reduced contents of cellulose and lignin. EC, TR, LA and SH decompose in a similar way. Denser wood samples, such as CA and TC, exhibit the highest thermal stability. Moreover at 450°C , weight loss is only 60 wt% for these two woods versus 90-100 wt% for all other species. The elevated contents of lignin are probably responsible of this behaviour. Decomposition continues up to 600°C . A comparison between the thermogravimetric curves at high temperature for TD and CA or TC shows a shift of almost $150\text{-}200^{\circ}\text{C}$ between them.

Pyrolysis in PCFC is carried out under nitrogen. Moreover, the sample size is very low and does not influence the heat release rate curves (no thermal inertia). Therefore, no effect of the density is expected. Figure 4 shows the curves for all wood species. Table 3 lists the main data. The peak of heat release rate appears in a narrow temperature range from 346 to 373°C and its intensity ranges from 80 to 130 W/g. Dorez et al. have obtained similar values for a set of ligno-cellulosic materials[20], including cellulose, hemicellulose, lignin and natural fibres. Nevertheless, total heat release is slightly higher for wood species (8.2 to 13.3 kJ/g versus 5.1 to 9.1 kJ/g in Dorez work). Note that LA is characterized by the lowest pHRR (80 W/g) and THR (8.2 kJ/g) but also by a shoulder at low temperature (around 250°C). On the contrary, CA exhibits the highest

pHRR (126 W/g) and THR (13.3 kJ/g). Obviously, the differences between the wood species are lower than in aerobic pyrolysis (in thermogravimetric experiments).

The main results of epiradiator test and LOI test are summarized in table 4. The initial ignition time increases with the density of the wood species (Figure 5). Only CA sample seems to exhibit a slightly lower TTI than expected. LOI values range from 21.1 to 26.5. It is noteworthy that the three densest species (LA, CA and TC) exhibit the highest values. Nevertheless, the relation between LOI and density is rather rough. In particular LA exhibits the highest LOI. This wood species seems also to be more self-extinguishing than the other ones. The flame persistence is only 9s versus 16-29s for the other species. On the other hand, the TD sample with lowest density obtained the worst results in both epiradiator and LOI tests. However, once again the correlation between the flame persistence and the density as well between LOI and flame persistence appears limited. No relation is observed between the weight loss and the other data, as total burning time (calculated from the number of ignitions and the average flame persistence) or LOI. These results show that despite wood density has an influence on the fire performance it cannot be taken as the only affecting factor.

Cone calorimeter is a powerful tool to study the flammability of samples. Figure 6 shows typical curves of CA and EC, respectively high and low-density species. All samples exhibit similar curves with two peaks of heat release rate. The second peak is usually assigned to the breakdown of a char layer or to the change from thick to thin thermal behaviour [21]. When the flame vanishes, heat release rate drops quickly up and stabilizes around 30-80 kW/m². This plateau corresponds to thermo-oxidation of the residue.

Main cone calorimeter data are listed in Tables 5 and 6. Sample weight varies from one wood species to the other one. This obviously implies changes in some data as total heat release in MJ/m², time to pHRR, time to plateau or flame out. For thermally thin samples, time-to-ignition or pHRR also depend on sample weight. Nevertheless, considering the duration of burning, the samples can be reasonably considered as thermally thick.

Time-to-ignition increases when density increases as observed for epiradiator test (Figure 7). This can be ascribed to lower thermal conductivity of less dense wood species [22]. Indeed, porosity limits the heat transfer from the surface to the sample bulk that allows a faster increase of the surface temperature. CA exhibits a quite unexpected low time-to-ignition as already suggested from epiradiator tests. Time-to-ignition in cone calorimeter and time-to-first ignition in epiradiator test are perfectly correlated (Figure 8). Time-to-ignition depends on a couple of parameters including the spectral distribution of the radiation, i.e. heat source and the heat absorbance of the sample. The low time-to-ignition of CA needs further investigation.

For each sample, the first peak of HRR is significantly lower than the second one (Figure 9). The first pHRR is in the range 120-145 kW/m² for all species except the densest one (TC – 199 kW/m²).

Concerning the second pHRR, two groups can be separated. CA and TC as denser species exhibit much higher pHRR (350-400 kW/m²) at significantly longer times. All other wood species can be gathered in a second group with pHRR mainly in the range 220-270 kW/m² (Figure 10).

Total heat release and effective heat of combustion were calculated for each sample at the beginning of plateau, i.e. before thermo-oxidation (pyrolysis is believed to be

anaerobic before the flame vanishes). Total heat release at the beginning of plateau is in the range 6.2-10.8 kJ/g, i.e. slightly lower (- 15%) than the values measured in PCFC (in anaerobic conditions). This gap can be assigned to an incomplete combustion in cone calorimeter test. CA and LA exhibit respectively the highest and the lowest total heat release as already observed in PCFC.

Even if there are some differences in residue content, this tendency is also observed for effective heat of combustion (see Table 5). LA has the lowest effective heat of combustion at the beginning of plateau while CA has the highest value. This evidences that the pyrolytic gases (and the compositions) are different between these wood species.

Residues at the beginning of the plateau range from 22.5 to 34.2 wt% but no clear tendency with density is observed (Table 5). Note that LA exhibits the highest residue content. During plateau, the residue content decreases continuously due to thermo-oxidation. This decrease is similar for all wood samples. Its mean value is around 2 wt% during the first 100s of plateau.

Another interesting point concerns the minimum reached by the heat release rate between the two pHRR. Indeed, it may be suggested that flame out can occur if the HRR decreases after pHRR1 below a threshold value. For all the wood samples tested in this article, it is not the case, and HRR starts increasing up to a second and intense pHRR. It can be noted that this minimum value of HRR ranges from 34 to 99 kW/m². No correlation with density is observed even if this value is significantly higher for the two densest samples. Once again, LA exhibits the lowest value, i.e. LA may be the closest sample to flame out.

Smoke production and CO/CO₂ ratio are other important data to assess the fire hazard. CO/CO₂ ratio is shown in figure 11 for CA and EC wood species. Similar curves are observed for all other species. The ratio is initially low with a small peak corresponding to the first pHRR1. Nevertheless, it increases sharply when thermo-oxidation starts. Indeed when flame vanishes, temperature in the zone above sample decreases and oxidation of partially oxidized gases released from aerobic pyrolysis becomes incomplete or even is stopped. Smoke production rate (SPR) is shown in figure 12 for CA and EC together with HRR curves. SPR curves exhibit two peaks for each wood species corresponding to the two peaks of heat release rate. Obviously, the smoke release increases when heat release increases. Nevertheless, the ratio between the first and the second peaks of SPR is most often lower than the ratio between the first and the second peaks of HRR. It means that the smoke release per released energy unit is more important during thermo-oxidation, in good agreement with the observed increase of CO/CO₂ ratio. Interestingly significant differences between wood species can be found even during anaerobic pyrolysis. Table 6 shows the total smoke production, total heat release and ratio between them at the beginning of the plateau. CA and TC exhibit significant higher values of both total smoke production (in m²/g) and smoke per released energy (in m²/kJ) confirming that the densest wood species have specific fire properties in comparison to other ones. The lowest values are observed for LA, confirming this wood species induces a lower fire hazard.

4. CONCLUSIONS

The assessment of the fire reaction of seven tropical wood species has been carried out using different fire tests with a particular attention to the role of density. The experimental methods developed have allowed us to compare different aspects.

In general, rather good relation is found between time-to-ignition and density this can be easily explained by the lower thermal conductivity of more porous materials. However, in some cases, other factors related to composition (inorganic compounds) and/or wood morphology must be taken into account because its significant influence in the fire behaviour. That is the case of *Lysiloma acapulcensis* (LA) species, which exhibits higher LOI and residue content, and lower THR and smoke release among all other studied wood species despite its medium density.

Denser wood species like *Cordia elaeagnoides* (CA) and *Tabebuia chrysantha* (TC) exhibit specific properties as slightly higher pHRR or higher smoke release. This is due to the greater mass of the denser wood samples.

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Table 1: Characteristics of different wood species

Code	Scientific name	Density kg/m ³	Moisture (%)
TD	<i>Tabebuia donnell-smithii</i>	448	8.9
EC	<i>Enterolobium cyclocarpum</i>	504	8.9
TR	<i>Tabebuia rosea</i>	604	9.0
SH	<i>Swietenia humilis</i>	655	9.9
LA	<i>Lysiloma acapulcensis</i>	685	9.2
CA	<i>Cordia elaeagnoides</i>	1130	7.3
TC	<i>Tabebuia chrysantha</i>	1234	8.3

Table 2: Summary of results of calcined residue analysis

Code	Calcined Residue (%)	Main elements
TD	0.6	P, Ca, K
EC	0.5	Ca, K
TR	0.5	Ca
SH	0.4	Ca
LA	1.3	K, S, Ca
CA	1.5	Ca
TC	2.8	Ca

Table 3: Values of pHRR, TpHRR and THR obtained with the PCFC tests.

Code	Density (kg/m ³)	pHRR (W/g)	TpHRR (°C)	THR (kJ/g)
TD	448	117	354	10.1
EC	504	106	365	10.0
TR	604	113	362	10.5
SH	655	107	366	9.5
LA	685	80	346	8.2
CA	1130	126	373	13.3
TC	1234	93	368	9.0

Table 4: Summary of results of flammability and LOI testing.

Code	Density (kg/m ³)	Initial ignition (s)	Number of ignitions	Avg. flame persistence (s)	Weight loss (g)	LOI (%)
TD	448	17	8	29	6.97	21.1
EC	504	17	12	16	4.6	24
TR	604	24	8	29	3.75	21.5
SH	655	28	14	16	4.77	23.1
LA	685	31	16	9	3.4	26.5
CA	1130	37	9	26	2.28	25.1
TC	1234	49	13	20	4.77	24

Table 5: Summary of results of cone calorimeter test.

Code	Density (kg/m ³)	TTI (s)	pHRR 1 (kW/m ²)	TpHRR1 (°C)	pHRR2 (kW/m ²)	TpHRR2 (s)
TD	448	38/36	124/116	45	248	330/325
EC	504	32/38	140/128	40/45	204/252	335/325
TR	604	56/57	125/128	70	240	395/365
SH	655	65/70	128/129	70/80	246/215	405/470
LA	685	75/68	128/162	80/75	256/288	450/390
CA	1130	87/98	150/137	100/120	348/341	645/595
TC	1234	133/155	212/186	145/165	409/369	630/625

Table 5: Summary of results of cone calorimeter test (continued).

Code	Density (kg/m ³)	THR before plateau (kJ/g)	EHC before plateau (kJ/g)	Residue before plateau (wt%)	Minimum HRR between pHRR (kW/m ²)
TD	448	8.6/9.3	10.6/12.6	18.6/26.4	59/61
EC	504	7.9	11.1/12.3	28.8/36	39/51
TR	604	9.8/8.5	12.1/10.5	19.3	66/68
SH	655	10.2/8.1	11.7/11.8	27/30.9	52/57
LA	685	5.6/6.7	8.2/10.6	31.9/36.5	29/39
CA	1130	10.8	13.9/14.0	22.3/22.9	92/103
TC	1234	10.3/8.5	14.2/11.7	27.2	93/105

Table 6: Summary of smoke results of cone calorimeter test

Code	Density (kg/m ³)	TSR (m ² /g)	THR (kJ/g)	Ratio TSR/THR (m ² /kJ)
TD	448	6.2-7.3E-02	8.6/9.3	7.2-7.9E-03
EC	504	6.1-7.0E-02	7.9	7.7-8.9E-03
TR	604	6.1-7.6E-02	9.8/8.5	7.8-9.0E-03
SH	655	4.7-5.3E-02	10.2/8.1	5.5-5.8E-03
LA	685	3.3-3.7E-02	5.6/6.7	5.5-6.6E-03
CA	1130	2.4-2.5E-01	10.8	2.3E-02
TC	1234	2.0-2.1E-01	10.3/8.5	2.0-2.4E-02

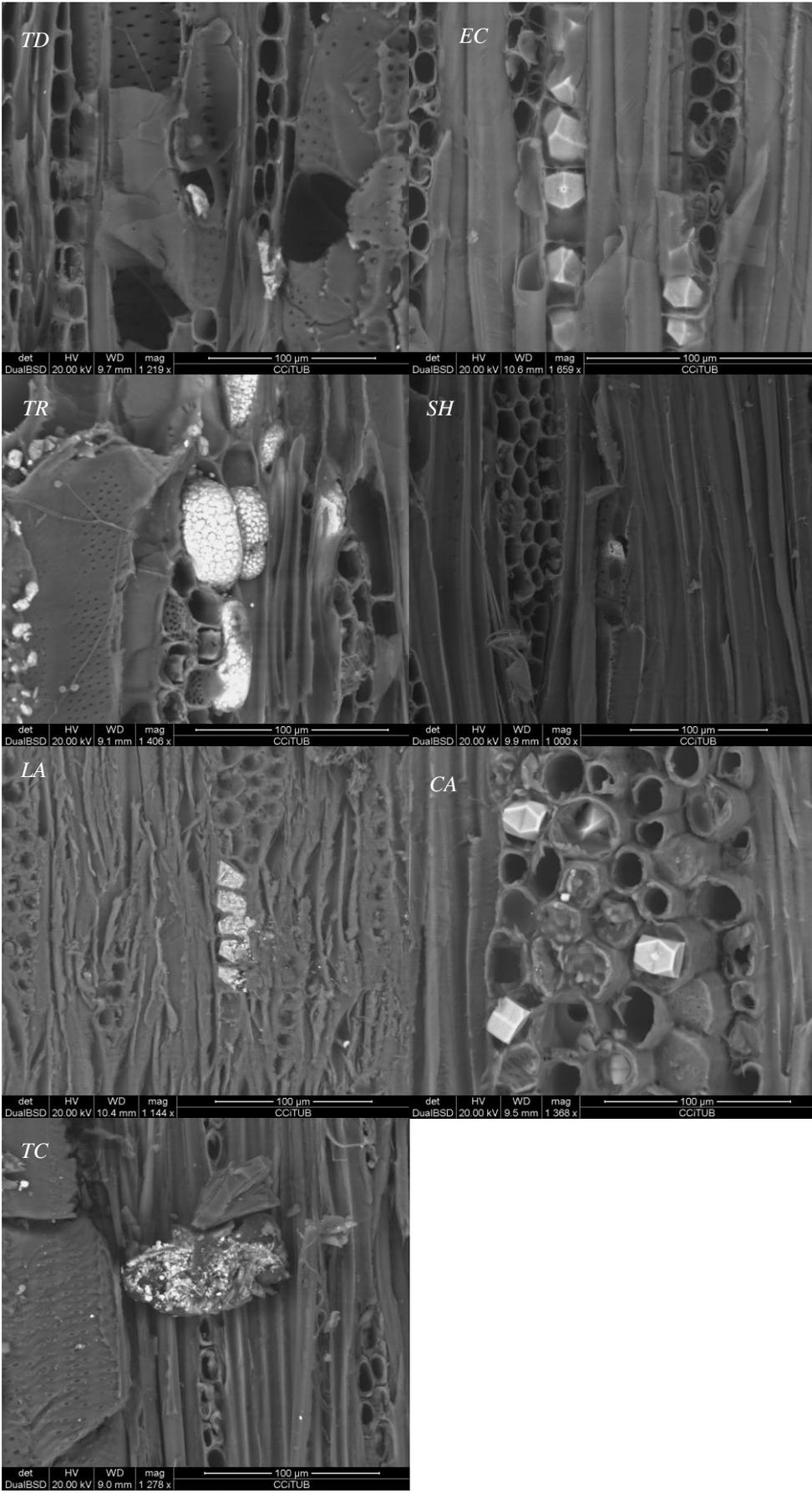


Figure 1. SEM morphology of wood samples

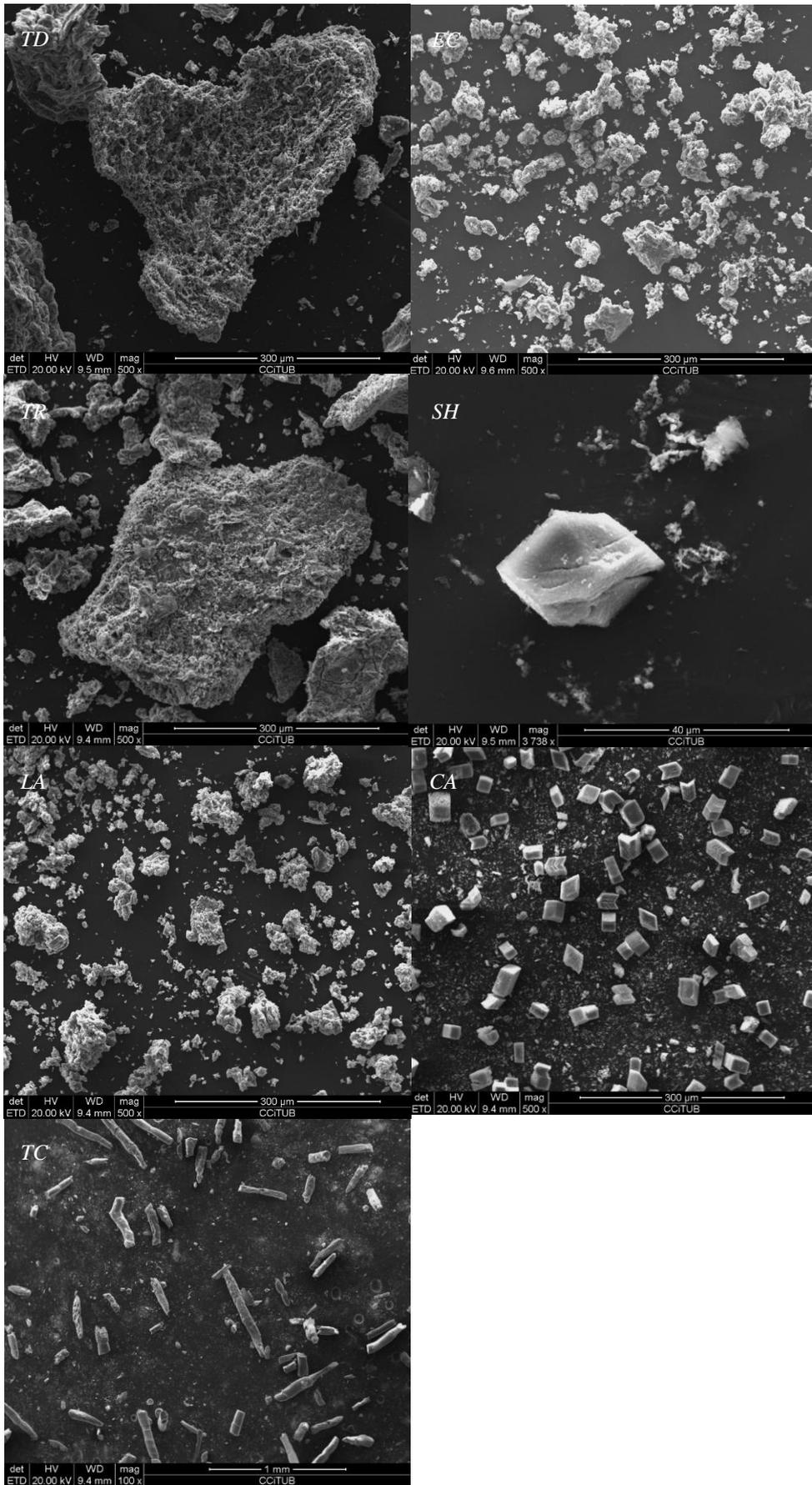


Figure 2. SEM images from the calcined residues.

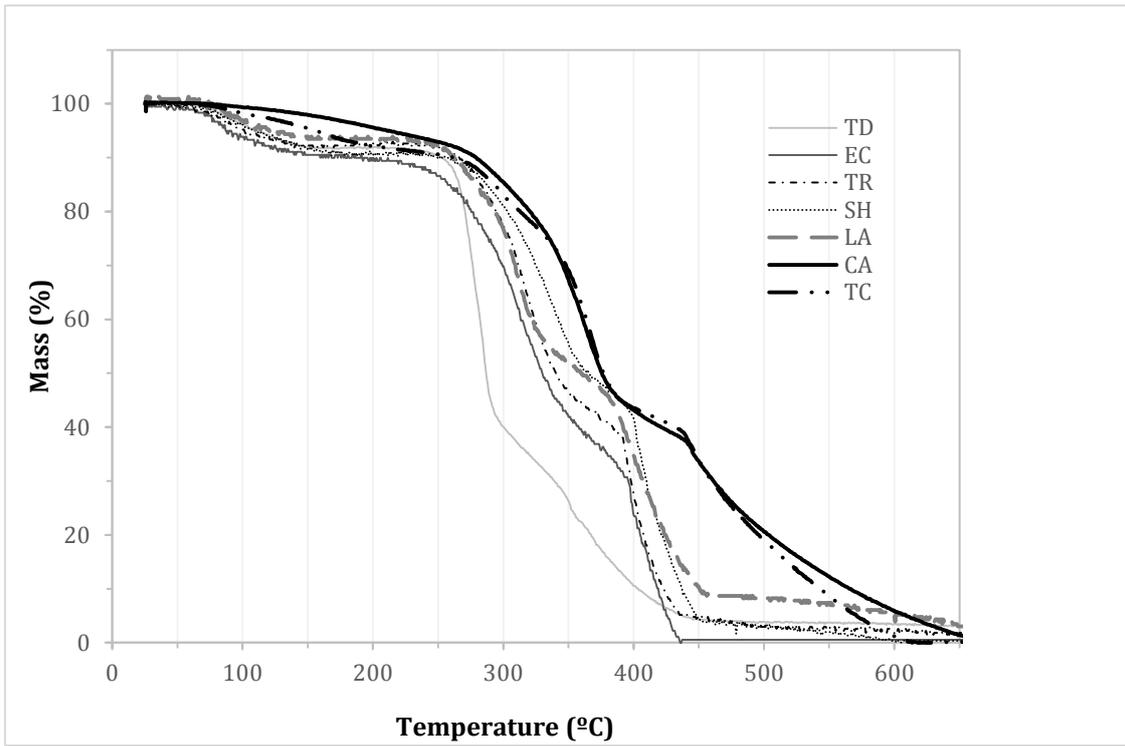


Figure 3. TGA curves of the seven hardwood species

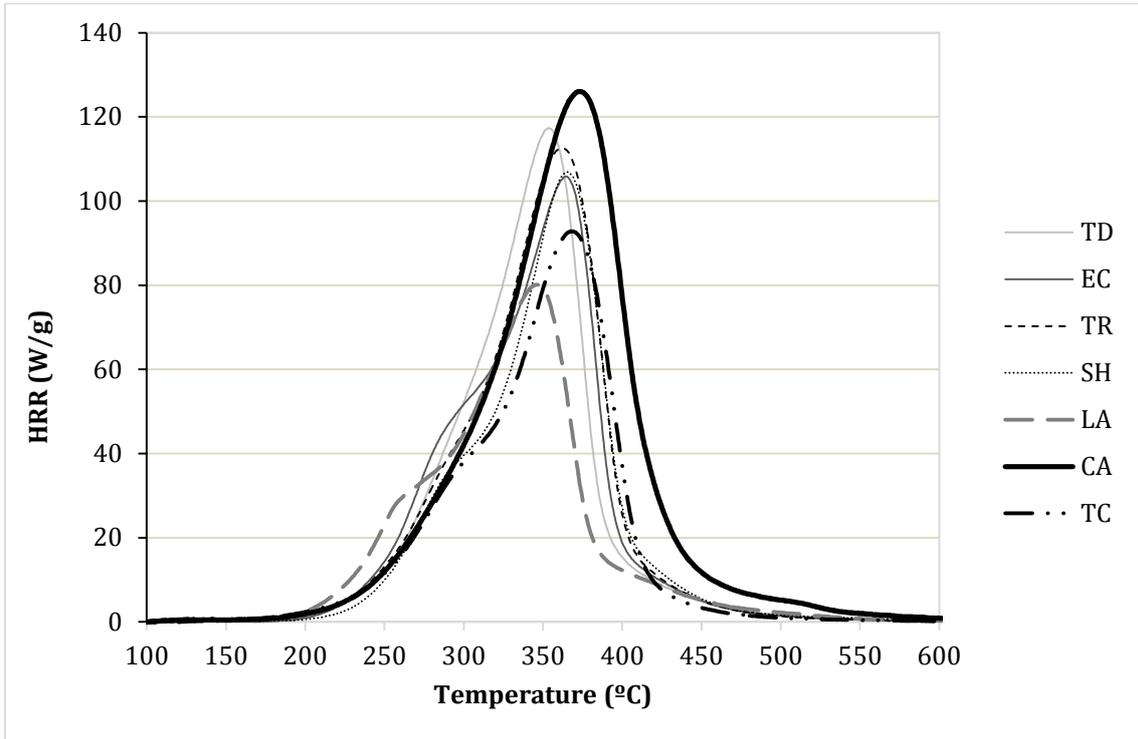


Figure 4. HRR versus temperature for all wood species in PCFC.

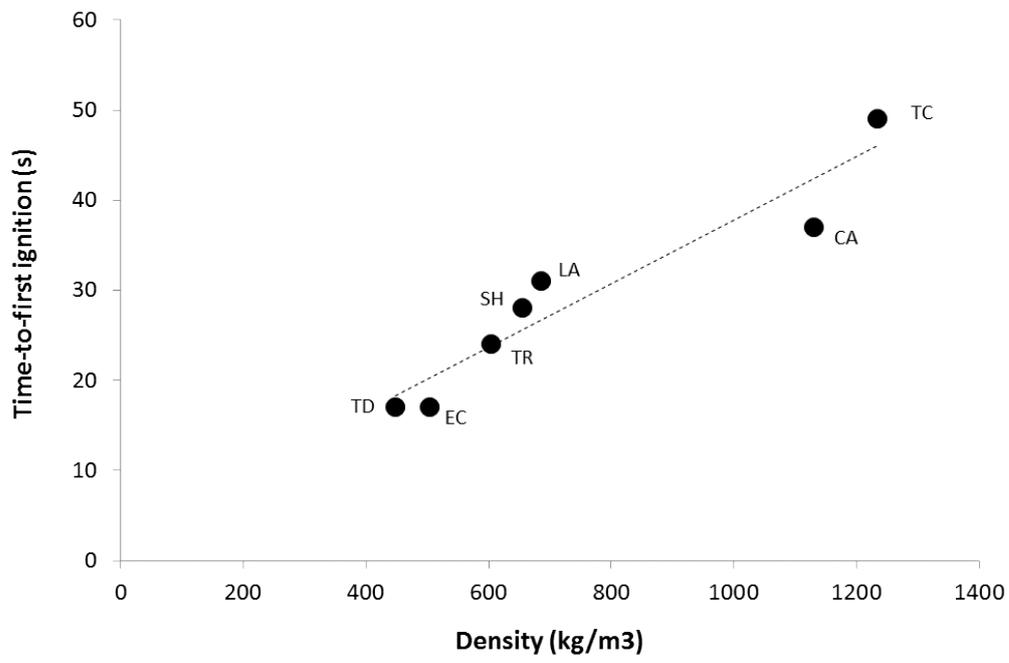


Figure 5. Time-to-first ignition versus density for all wood species according to epiradiator test.

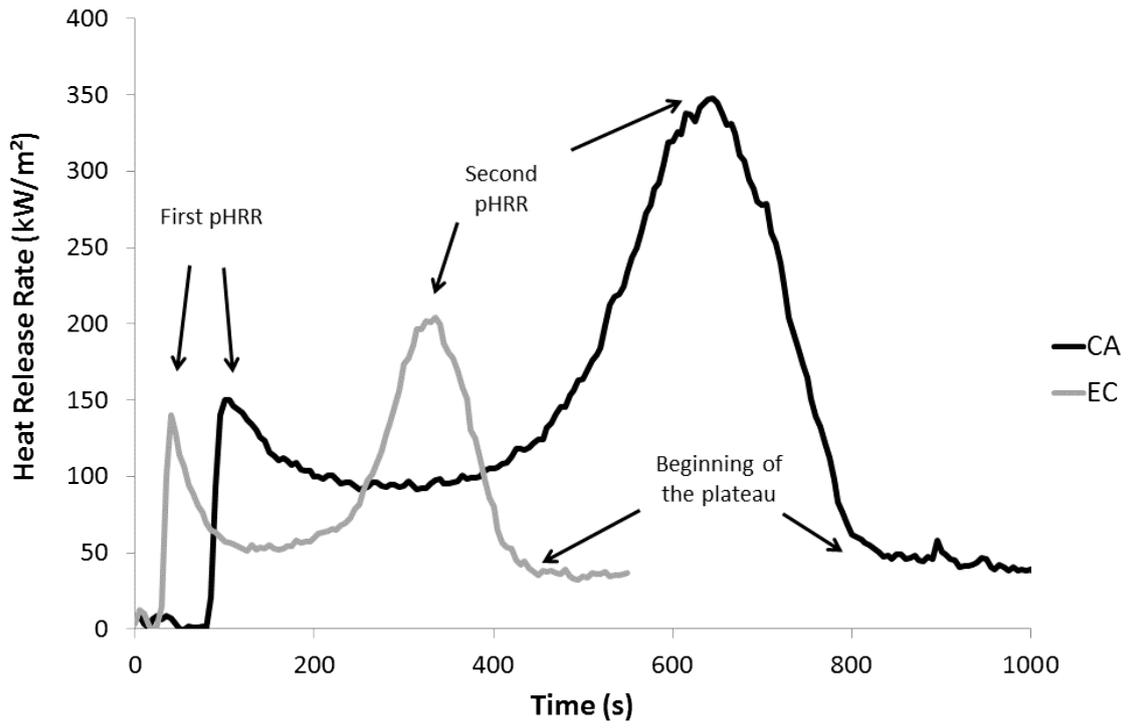


Figure 6. HRR versus time for samples CA and EC in cone calorimeter tests.

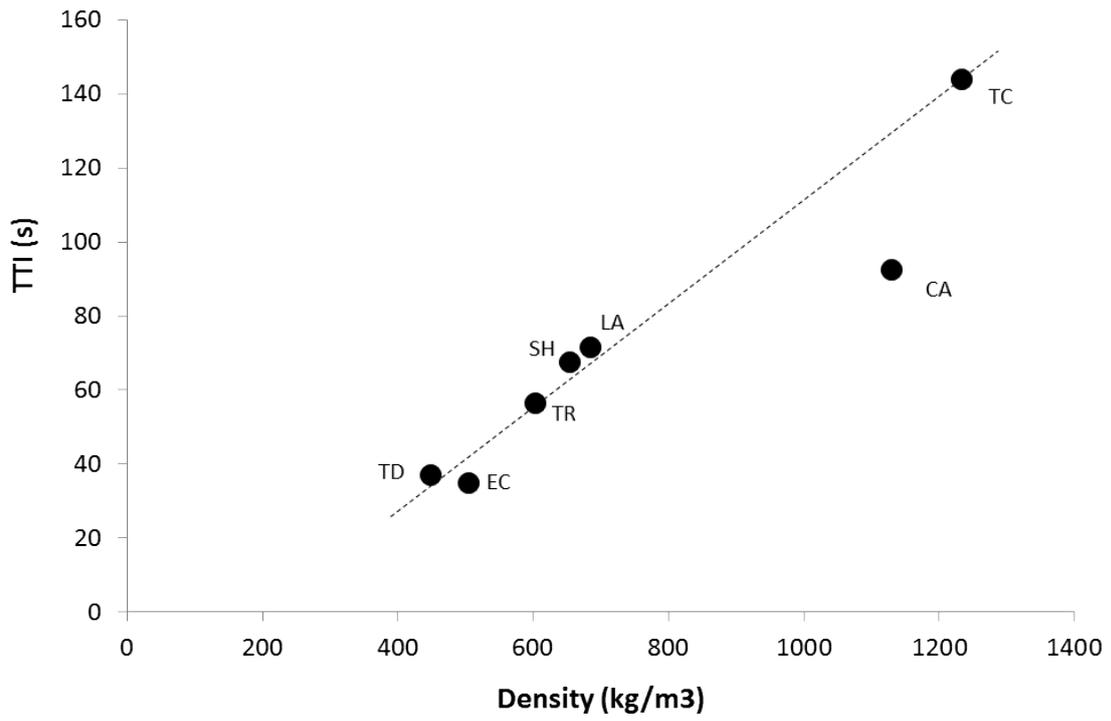


Figure 7. Time-to-ignition versus density for all wood samples in cone calorimeter tests.

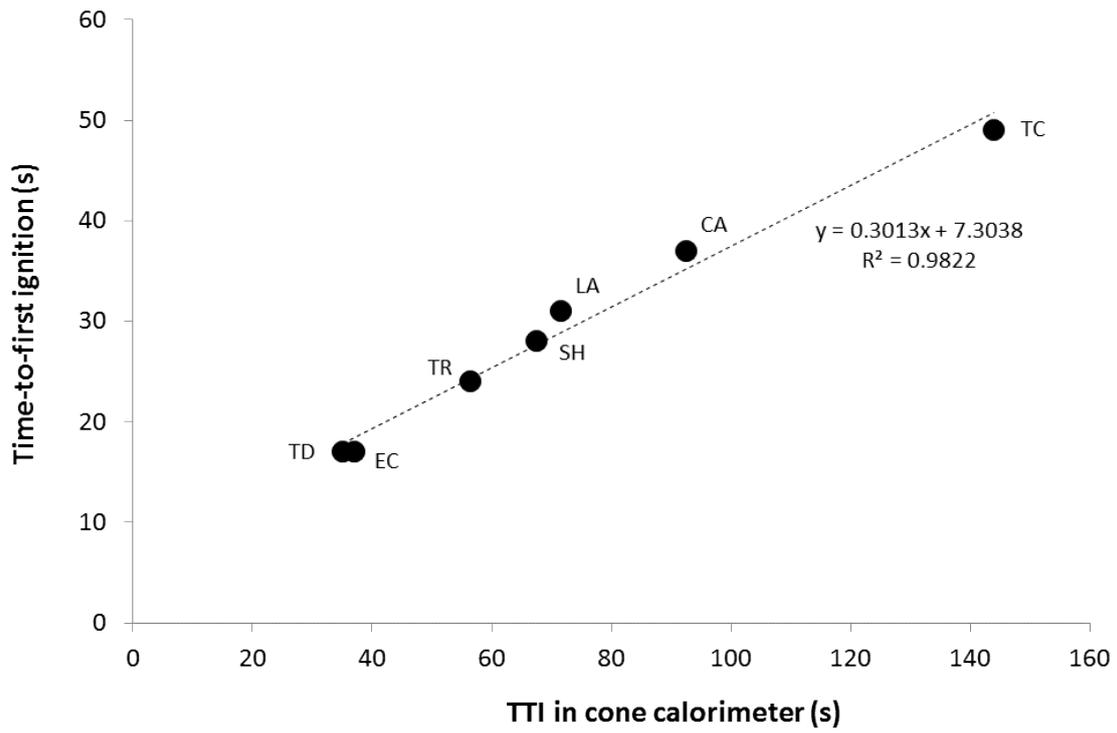


Figure 8. Time-to-first ignition from epiradiator test versus time-to-ignition from cone calorimeter test for all wood samples.

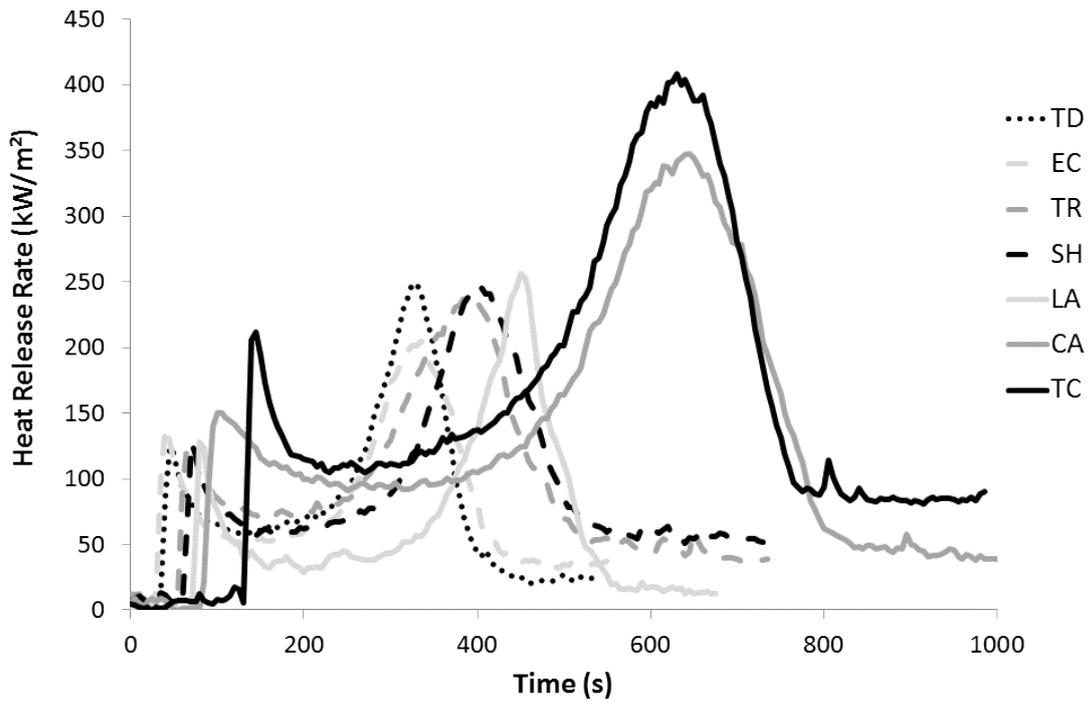


Figure 9. HRR versus time of all the hardwood samples in cone calorimeter tests.

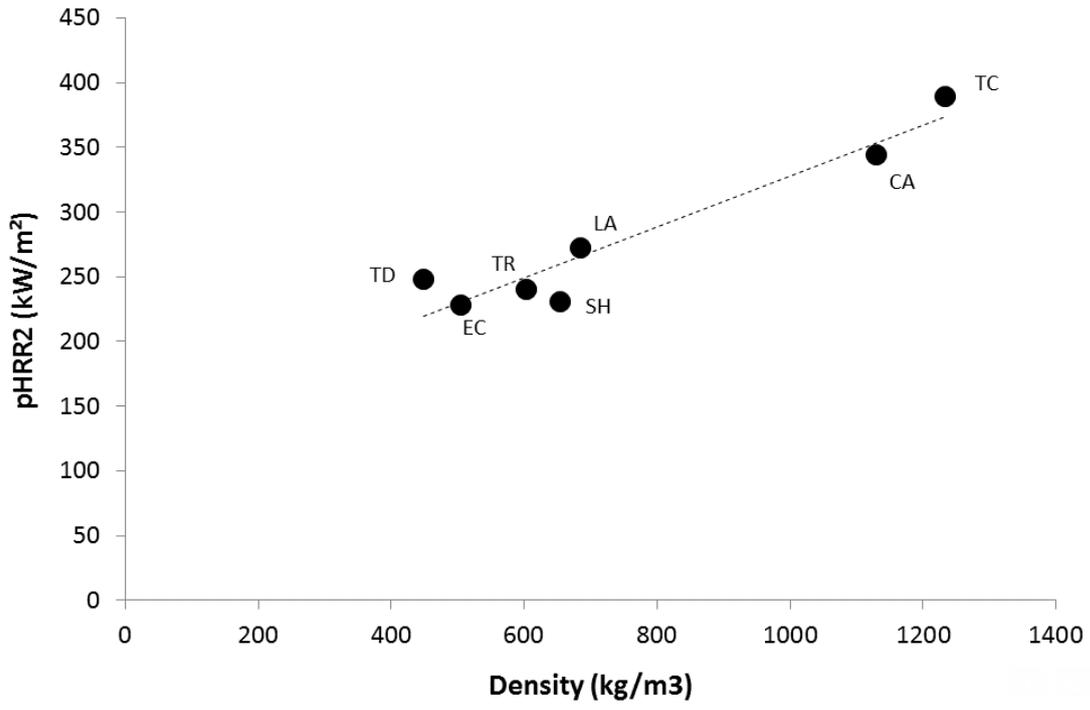


Figure 10. Second peak of heat release rate versus density for all wood samples.

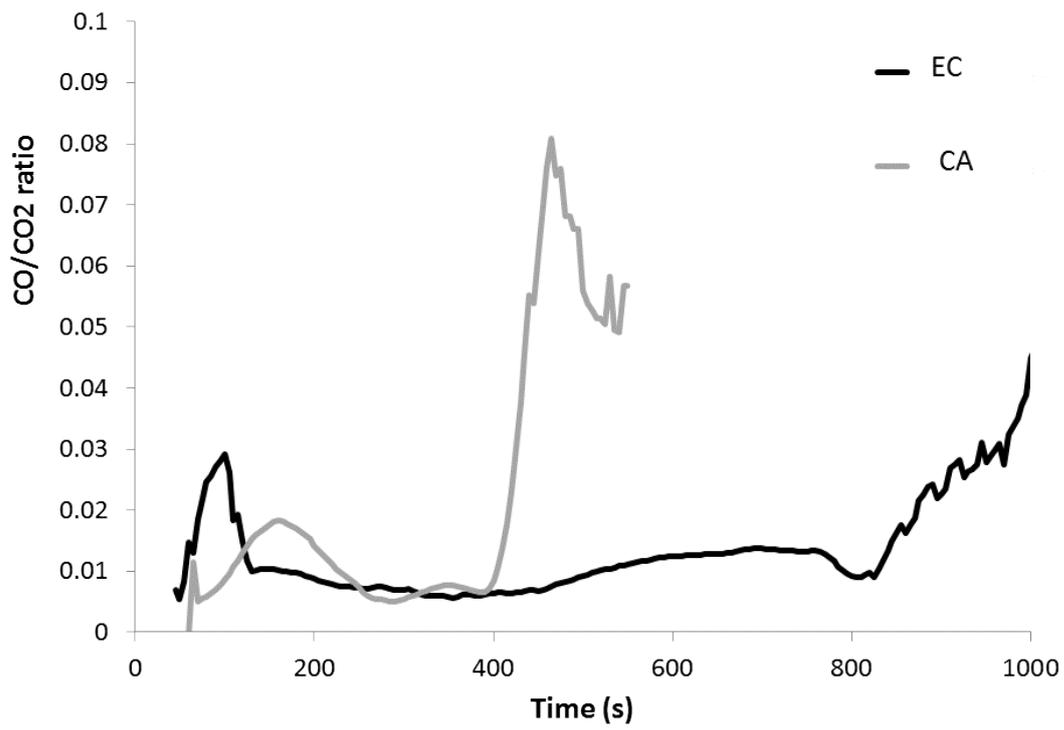


Figure 11. CO/CO₂ ratio versus time for two wood samples in cone calorimeter tests.

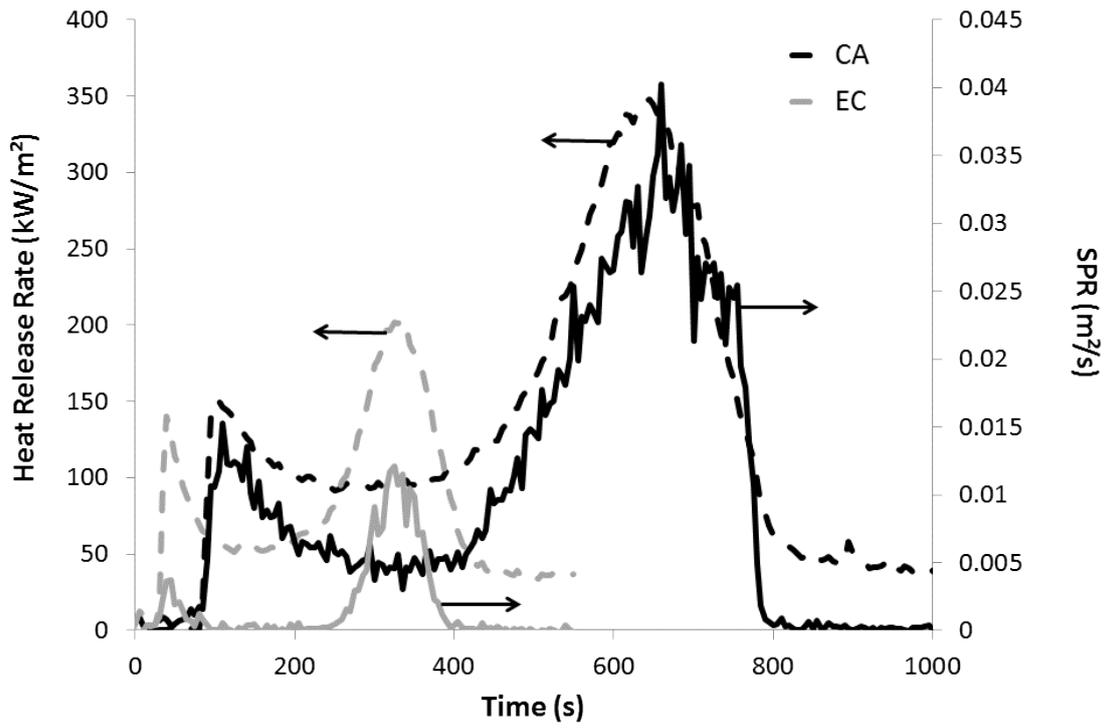


Figure 12. Smoke production rate and HRR versus time for two wood samples in cone calorimeter tests.