A method for measuring the relative particle fire hazard properties of forest species

S. Liodakis *, D. Vorisis, I.P. Agiovlasitis

Department of Chemical Engineering, Sector 1, National Technical University of Athens (NTUA),
9 Iroon Polytechniou Str., 15773 Athens, Greece

Abstract

A simple method was developed to determine the relative particle foliar flammability and heat content of forest fuels. The apparatus used was similar to the one described by the 79/831 European Commission Directive for measuring the relative self-ignition temperature (minimum ambient temperature at which a certain volume of a substance ignites under defined conditions). The forest materials examined were: *Cupressus sempervirens* (Italian cypress), *Olea europaea* (Olive tree), *Pinus brutia* (Calabrian pine), *Pinus halepensis* (Aleppo pine), *Pistacia lentiscus* (Mastic tree), *Quercus coccifera* (Holly oak). These species are very common in the Mediterranean region and frequently devastated by forest fires. The relative data determined (self-ignition temperature, ignition delay times, combustion duration, etc.) provide a simple screening test on the fire hazard properties of forest species, which could be used for comparison purposes. Among the forest species examined, *Pinus brutia* had the highest relative ignition temperature and the longest ignition delay time, while *Pistacia lentiscus* had the lowest and shortest ones. Also, *Quercus coccifera* and *Pistacia lentiscus* had the highest heat content and *Olea europaea* the lowest one. Finally, *Cupressus sempervirens* was the most combustible and *Pinus halepensis* the most sustainable fuel.

1. Introduction

Wild fires burn thousands of hectares all over the world each year. The flammability study of forest species is very important to forest fire management, i.e. facilitates the selection of the appropriate afforestation species for reducing wildfire danger.

The thermal degradation of forest fuels can be simplified by considering two consecutive steps. The first is the *pyrolysis*, which is endothermic process and breaks down the forest matter into low molecular mass gases (volatiles), tars, carbonaceous char and mineral ash. The second is the burning process, is exothermic and is known as *combustion*. *Ignition* is the transition between the first and second step.

Various techniques have been successfully used for the thermal degradation study of forest fuels such as thermal analysis, i.e. differential scanning calorimetry (DSC) and thermogravimetry (TG) [1–4], hyphenated, i.e. direct inlet–mass spectrometry (DI–MS), pyrolysis–gas chromatography–flame ionization detector (Py–GC–FID), pyrolysis–gas chromatography–mass spectrometry detector (Py–GC–MSD) and thermogravimetry–mass spectrometry (TG–MS) [5–7] and electrochemical analysis [8].

However, the small quantities of samples used in the above analytical methods and the rapid removal of pyrolysis and combustion products can lead to an erroneous interpretation in terms of forest fuel flammability performance in field conditions. Therefore, the information provided by analytical techniques should be supported by other laboratory-scale flammability tests [9–14].

The flammability can be defined as the easiness of a material to catch fire both spontaneously or through exposure

* Corresponding author. Tel.: +30 210 7723116; fax: +30 210 7723188.
E-mail address: liodakis@central.ntua.gr (S. Liodakis).

to certain ambiences. The flammability of forest species, according to Anderson aspects [15], includes three components: ignitability, combustibility and sustainability. The ignitability determines how easily the fuel ignites. Combustibility is the rate of burn after ignition. Sustainability counts the stability of burning rate, or how well the fuel continues to burn.

The flammability of natural fuels is a complex phenomenon, which cannot be adequately assessed in the field due to the influence of many variable environmental factors that can neither be controlled nor monitored [16,17]. The laboratory tests provide the ability to investigate the flammability of various fuels as it is affected by their chemical constitution and hydric condition, excluding all external factors.

Moreover, most laboratory experimental studies employ a single fuel property to represent flammability. Among them the most popular are the temperature of ignition, the time to ignition (ignition delay time) and the heat content.

Two types of ignition are distinguished: pilot and spontaneous. In piloted ignition flaming is initiated in a flammable vapour–air mixture by a pilot, such as an electrical spark or a flame. In spontaneous ignition flaming is developed spontaneously within the flammable vapour–air mixture. The ignition temperature can be defined either as: (1) the minimum surface temperature at which the flow of volatiles is sufficient for sustained flaming; or as (2) the minimum temperature of furnace sufficient for a specimen put therein to ignite [18].

A number of tests have been proposed for the determination of the ignition temperature in laboratory-scale, i.e. pulse-ignition test [19]. Among them, the most common is the *Setchkin ignition* test [20,21]. This is based on the determination of the lowest initial temperature of air passing around the specimen at, which a sufficient amount of combustible gas is evolved to be ignited in the presence or absence of an external pilot flame. Lohrer et al. [22] have examined various criteria for evaluating spontaneous ignition, as the mass loss, the discoloration and the temperature increase of samples ignited. Thermal analysis has been also applied to determine the self-ignition of solid fuels, such as wood, peat, lignite coals, brown coals, anthracite [23–27], biomass [18] and explosives [28,29].

Sustainability is the ability of the fuel to carry fire through it to the next fuel. There is a carry-over of energy from one fuel type into the next. Under moderate burning conditions, a fire may carry into the next fuel type, but that fuel type may not be able to sustain combustion. Thus, sustainability is related to the rate of fire spread. Mak [30] has quantitatively measured the foliar sustainability and ignitability of 10 selected Australian trees and shrubs using the Limiting Oxygen Index (LOI) method.

Another important factor related to the flammability of forest species is their heat content (heat of combustion), which can be measured by static bomb calorimeter in an oxygen atmosphere or by differential scanning calorimetry (DSC). Rodriguez-Anon et al. [31–35] and Nunez-Regueira et al.

[36–41] have determined the high and low heating values and the flammabilities of various forest species during the seasons of the year. The flammability experiments were performed, following the standard UNE-23-721 method, using a standard epiradiator of 500 W constant nominal power. Flammability values were obtained according to the tables proposed by Valette [36].

The objective of the present work is to develop a simple method for evaluating the relative flammability and heat content properties of forest species, using a specifically designed apparatus, under precisely controlled temperature and static air atmosphere conditions. The tests were performed after reducing plant leaves into a fine, uniform substance (particle foliar flammability). Thus, the study eliminates the influence of plant structure and the external characteristics of combustion (i.e. weather, climate and location) and counts the intrinsic components of the fuel (i.e. chemical composition). The main purpose of this study is to find the relative fire hazard properties of some very common forest species in the Mediterranean region and to rank them into categories. In addition to correlate this study with the development of wildfire retardants.

2. Experimental

2.1. Sampling data collection

The sampling of the leaves and needles of forest materials took place in the area Kalopoula of the mountain Ymittos near Athens. All samples were mature foliage, collected after a long drought period in order to avoid moisture effects. According to previous reports [42,43], the composition of a forest species depends on various factors: part of plant combusted (bark, wood, leaves), plant age, type of soil, climate, etc. As a consequence, a special care was taken to ensure that the bulk samples collected are representative to the forest species examined. The following data sheet (Table 1) shows some details for the conditions under which the sampling of forest fuels took place.

2.2. Sample preparation

The foliage forest samples gathered were placed into firmly closed polyethylene bags, brought immediately to the laboratory and dried into a vacuum oven for 24 h under pressure of 10 Torr and temperature of 60 °C.

The dried samples were ground and the following size fractions were selected: (1) $d > 830 \,\mu$m; (2) $830 \,\mu$m $> d > 500 \,\mu$m; (3) $500 \,\mu$m $> d > 315 \,\mu$m; (4) $315 \,\mu$m $> d > 200 \,\mu$m; (5) $200 \,\mu$m $> d > 90 \,\mu$m; and (6) $90 \,\mu$m $> d$, where d is the particle size (sieve opening).

The fractions 1 and 6 were rejected and the others were placed into a conditioning box, set at temperature of 32 °C and relative humidity of 12.5%. The equilibrium moisture content of forest samples tested was found 2.8% by linear insertion,

Table 1
Sample collection data

Date	18 October 2003
Local time	16:00–17:00
Position	Kalopoula, Ymmitos
Geographical coordinates	37° 58′ 13″N
	23° 48′ 20″E
Altitude	487 m
Exposition	W–NW
Average inclination	70%
Dominated rock	Limestone
Species	*Cupressus sempervirens* (Funeral cypres)
	Olea europaea (Olive tree)
	Pinus brutia (Calabrian pine)
	Pinus halepensis (Aleppo pine)
	Pistacia lentiscus (Mastic tree)
	Quercus coccifera (Holly oak)
Remarks	1. The geographical coordinates and the altitude where found by GPS and the exposition with a compass.
	2. The sampling area was 50 m around the above-mentioned geographical coordinates.

according to the tables given in the standard method ASTM D 4933 [44].

2.3. Apparatus for relative flammability and heat content measurements

The apparatus used for the relative particle foliar flammability and heat content measurements shown in Fig. 1 was designed according to 79/831 European Commission Directive [45]. The furnace chamber was vertical cylindrical with internal diameter 38 mm and length 300 mm. It was heated by coil electric resistance heaters, which are insulated with ceramic material and consumed electrical power 1000 W. Temperature control accuracy was ±2 °C in the
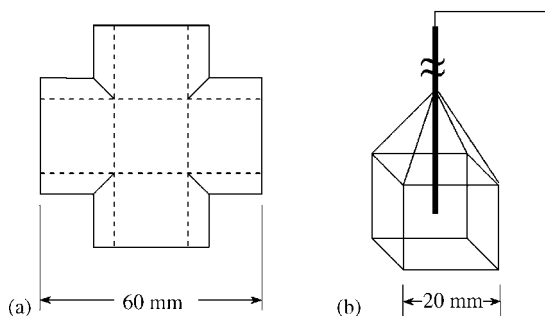

Fig. 2. (a) Pattern of 20 mm cubic sample holder. (b). The position of thermocouple in the sample holder.

range ambient −1200 °C, whereas the temperature uniformity was ±5 °C at 1000 °C. Also, a precise program system was used to control the heating rate between 1 and 100 °C min^{-1}. The furnace had a natural air circulation and an explosion relief system. A set of three Chromel/Alumel thermocouples, 0.05 mm diameter, were used to monitor temperatures at various points with an accuracy higher than 0.3 °C and a response time of 2–4 ms. One thermocouple was inserted into the sample holder, the other into the furnace space and the third into the atmosphere to monitor the sample, oven and ambient temperature, respectively. An analog thermocouple input module, Advantech model Adam-4018, stored up all temperature measurements, taken every 0.01–5 min, and converted them into digital for data handling.

The sample holder was made from stainless wire mesh with 0.045 mm openings, cut according to the pattern shown in Fig. 2a. Then, the mesh is folded and secured with wire into open topped cubes. The cube was filled with forest species to be tested and was tapped gently adding more of the sample until the cube was completely full. The sample was then suspended in the center of the oven at room temperature. One thermocouple was placed at the center of the cube and
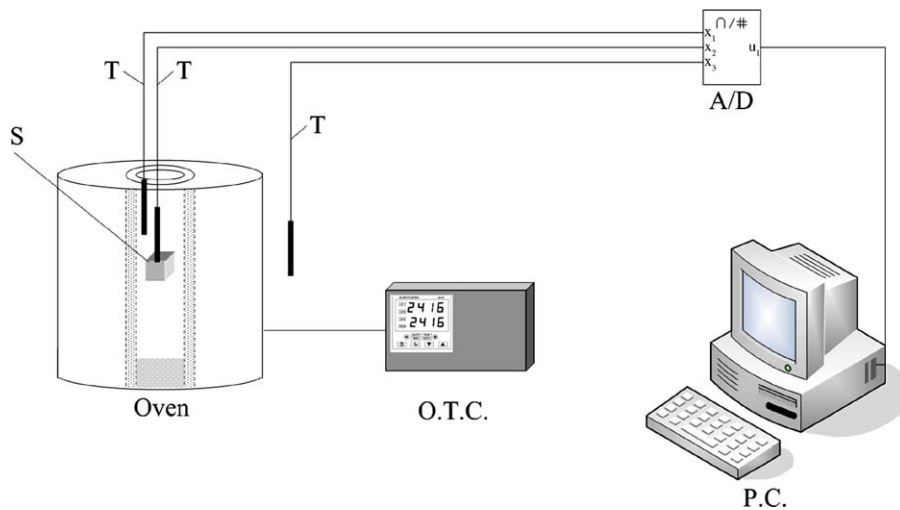

Fig. 1. Schematic diagram of the apparatus used for testing the relative flammability of forest species. S: sample holder; T: thermocouple; O.T.C.: oven temperature controller; A/D: analog/digital controller; P.C.: personal computer and recorder.
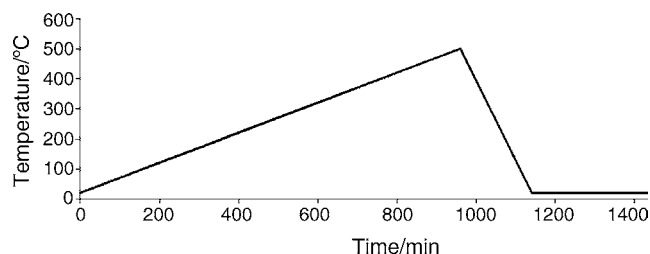
Fig. 3. Temperature program mode used for the flammability tests of forest fuels: heating from room to 500 °C with a constant rate of 0.5 °C min^{-1} and then cooling to room temperature in 180 min.

another between the cube and the oven wall to record the oven temperature (Fig. 2b).

2.4. Procedure for relative flammability and heat content measurements

The temperature of the oven and sample were recorded every 5 s, while the temperature of the oven was increased to 500 °C, at a rate of 0.5 °C min^{-1} (Fig. 3). The temperature of the oven at which the sample temperature reaches 400 °C is defined as relative self-ignition temperature and the respective time as relative ignition delay time (Fig. 4). By monitoring the forest sample temperature, the temperature profiles were recorded. From their graphical presentations the combustion duration and the relative rate of combustion were determined, two properties that can be related to the combustibility and sustainability, while the peak temperatures areas measured can be related to the heat content of forest samples.

The energy content of forest fuels was also determined by a Parr Instruments Company plain jacket bomb calorimeter, model 1341.
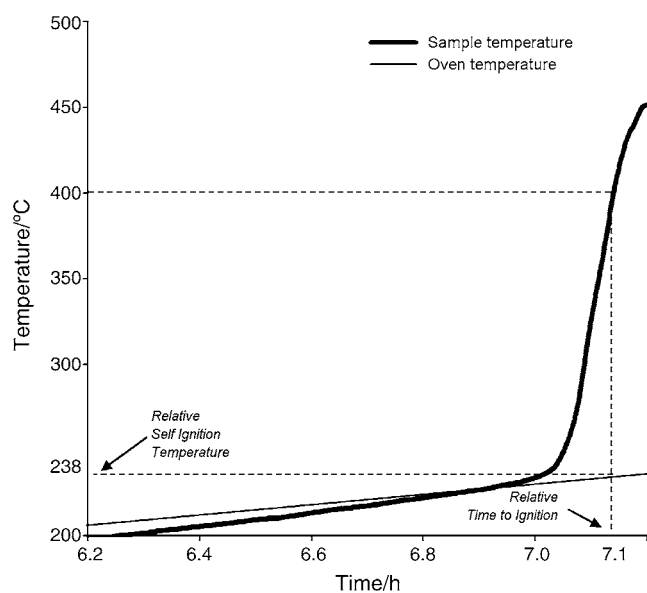

Fig. 4. A typical temperature profile for determining the ignition data of forest species.

3. Results and discussion

Fig. 5 shows the influence of particle size on the relative self-ignition temperature. Three replicate measurements were made for each particle size fraction studied: 90–200 μm, 200–315 μm, 315–500 μm, 500–830 μm. This study showed that the most consistent data were obtained with the fraction 200–315 μm, with relative standard deviation lower than 3% (Fig. 6). This particle size fraction was chosen for the flammability and heat content tests.

Fig. 7 shows the sample temperature–time profiles of forest species tested under the heating program mode described in Fig. 3. Based on these graphs, the relative self-ignition temperatures and relative times to ignition were determined (Table 2). The self-ignition temperatures and time to ignition values are related to the pilot ignitability, mainly involved in real forest fires. According to the literature, the fuels with high self-ignition temperatures and ignition delay times have low flash point values. Thus, they are the most ignitable fuels and they burn easily by flame [46].

Pinus brutia which has the highest values of relative self-ignition temperature (242 °C) and relative ignition delay time (7.39 h) is the most ignitable fuel, while *Pistacia lentiscus*
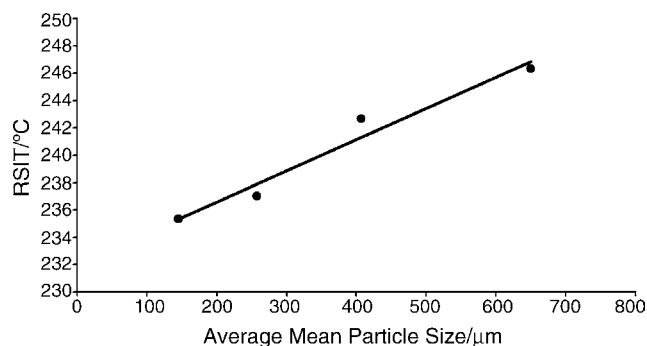

Fig. 5. The effect of particle size on the RSIT values of *Pinus halepensis*. RSIT: relative self-ignition temperature.
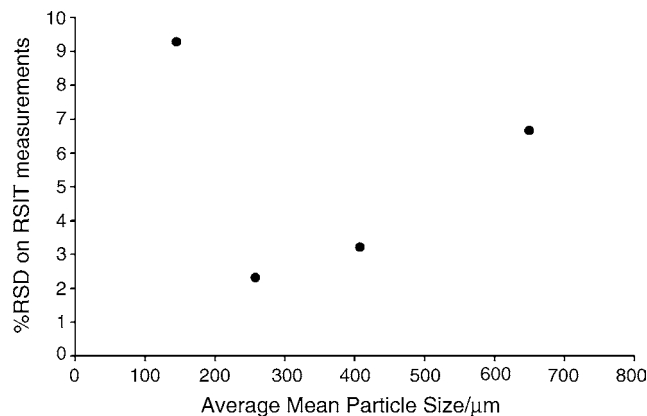

Fig. 6. The influence of sample particle size on the reproducibility of the relative self-ignition temperature (RSIT) measurements. RSD: relative standard deviation.
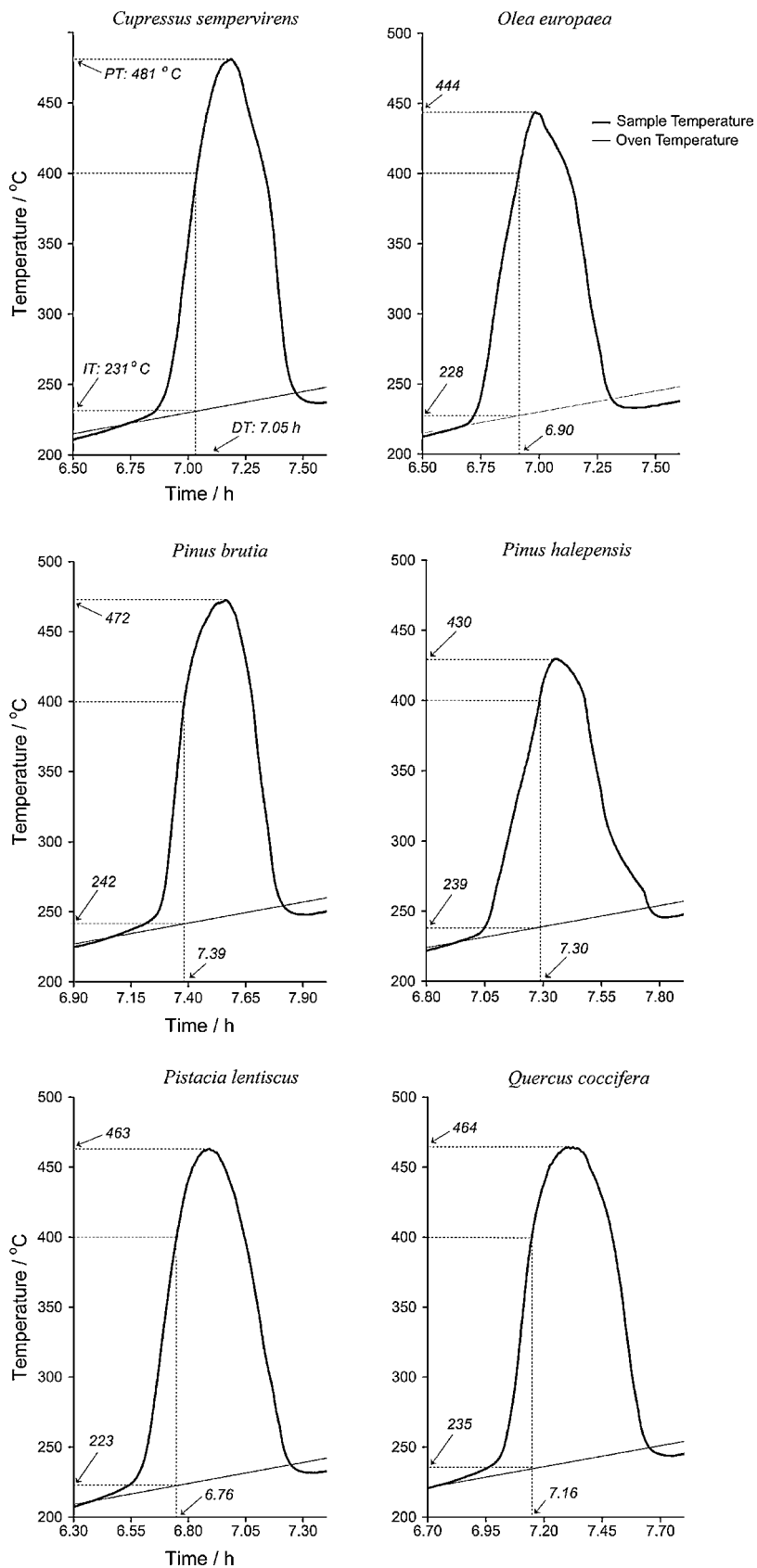
Fig. 7. Temperature profiles of forest species by applied a specific heating mode program. IT: (relative self-) ignition temperature; PT: peak temperature; DT: (relative ignition) delay time.

which has the lowest values (222 °C, 6.76 h) is the least ignitable one.

In addition, the maximum sample temperature (T_{max} or peak temperature) and peak area were recorded (Table 2), based on the temperature profile diagrams (Fig. 7). These data are related to the sustainability of fuel (the stability of the burning rate or the ability to sustain the fire once ignited).

The fraction of the temperature peak area/peak temperature was found proportional to its energy content (higher heating value, HHV), measured by a static bomb calorimeter, when reflecting the volume contained in the sample holder, giving a R^2 value of 0.91 (Fig. 8). Thus, *Quercus coccifera* and *Pistacia lentiscus* have the maximum heat content and *Olea europaea* the minimum one.
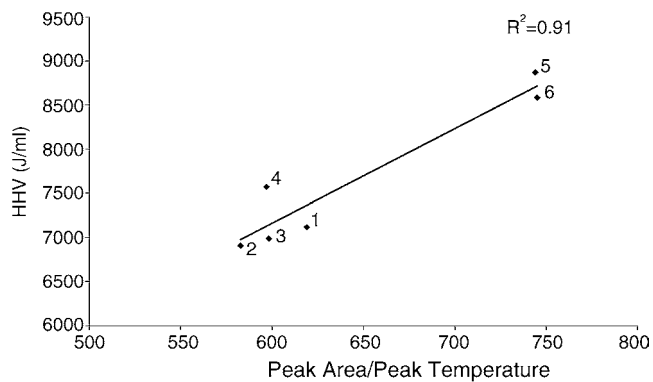

Fig. 8. Peak temperature area versus heat content of forest species. (1) *Cupressus sempervirens*, (2) *Olea europaea*, (3) *Pinus brutia*, (4) *Pinus halepensis*, (5) *Pistacia lentiscus*, (6) *Quercus coccifera*.
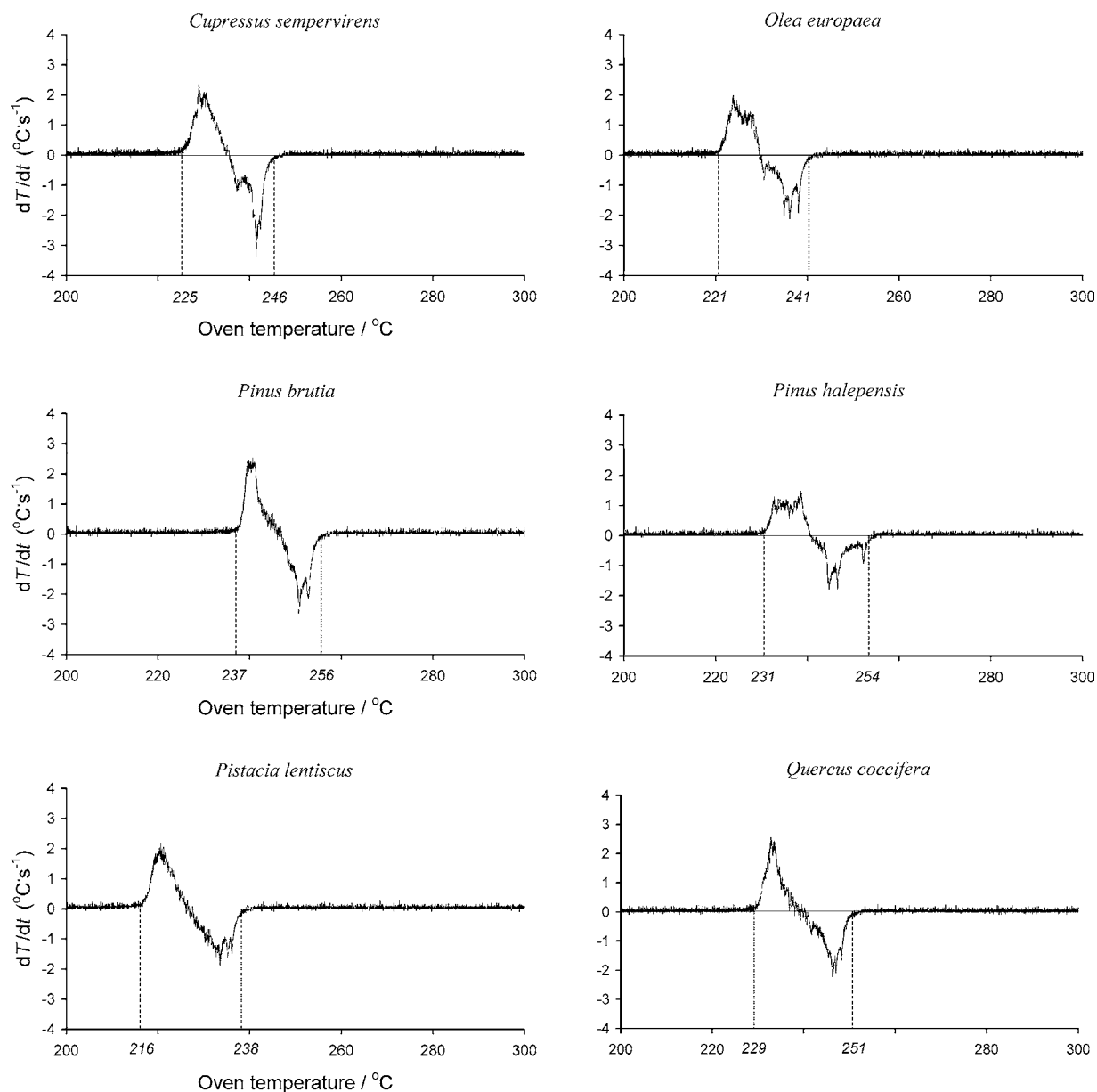

Fig. 9. First derivates curves of forest samples temperature profiles.

Table 2
Relative flammability and heat content data of forest species

Forest species	Self-ignition temperature (°C)	Relative time to ignition (h)	Peak temperature (°C)	Peak area arbitrary units	Combustion duration (min)	Initial/final combustion temperature (°C)	HHV (J/g)
Cupressus sempervirens	231	7.05	481	298	40.8	225/246	21,376
Olea europaea	228	6.92	444	259	39.2	221/241	21,933
Pinus brutia	242	7.39	472	282	37.2	237/256	20,677
Pinus halepensis	239	7.30	430	257	45.6	231/254	21,698
Pistacia lentiscus	222	6.76	463	344	44.1	216/238	21,531
Quercus coccifera	235	7.16	464	346	43.1	229/251	20,058

Fig. 9 presents the first derivative of sample temperature versus oven temperature. The first derivative is approximated as $\Delta T/\Delta t$, where ΔT is the change in sample temperature between successive measurements and Δt the time interval of 5 s. Based in these graphs the initial and final combustion temperature and the combustion duration were determined. The combustion duration is related to the combustibility (the rate of burn or the fire intensity) as well with the sustainability of fuel (the ability to sustain fire), with the most combustible fuels having shorter combustion duration and the most sustainable having longer ones. Both sustainability and combustibility are related to the fire spread.

Combining the experimental data given in Table 2, we found that the fraction peak area/combustion duration and peak temperature are correlated, with an R^2 value of 0.65 (Fig. 10). These parameters possibly measure the same property of forest species, likely combustibility and sustainability. Thus, the most combustible fuels have high values (right side of diagram) and the most sustainable ones have low values (left side of diagram). Fuels combining equally good combustibility and sustainability should be in the center and upper part of the diagram. This is in perfect agreement with the aforementioned observation about the forest fuels that have the higher heat content, *Quercus coccifera* and *Pistacia lentiscus* (Fig. 8).
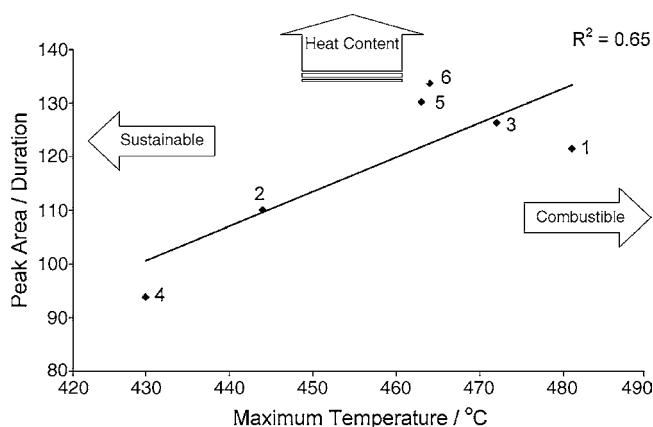

Fig. 10. Fraction of peak area/combustion duration versus peak temperature for estimating the combustibility and sustainability of forest species. (1) *Cupressus sempervirens*, (2) *Olea europaea*, (3) *Pinus brutia*, (4) *Pinus halepensis*, (5) *Pistacia lentiscus*, (6) *Quercus coccifera*.

4. Conclusions

The present work is focused on the development of a simple laboratory method for measuring the relative flammability of forest species. The experimental apparatus constructed for this purpose measures various hazard properties (peak area, peak temperature, combustion duration, etc.) by monitoring the sample temperature under specific heating mode conditions. These properties were related to the ignitability, combustibility, sustainability and heat content of forest fuels.

The most interesting conclusions drawn from our experimental study on six very common Mediterranean forest species are:

(1) The data obtained refer to particle level measurements in order to eliminate the influence of external factors (i.e. weather, climate, moisture content, particle size, etc.).
(2) The simple, low cost, apparatus developed measures relative properties. It has the potential to be used as a screening device, comparing the fire hazard properties of forest species and ranking them into categories of similar properties.
(3) Among the forest species examined, *Pinus brutia* has the highest relative ignition temperature and *Pistacia lentiscus* the lowest one. *Quercus coccifera* and *Pistacia lentiscus* have the highest heat content and *Olea europaea* the lowest one. Finally, *Cupressus sempervirens* is the most combustible fuel and *Pinus halepensis* the most sustainable fuel. These results obtained are in good agreement with previous research works [16,18].
(4) Further work should be carried out on relating flammability data with forest species chemical composition, external conditions and fire retardant additives. These data can be evaluated for studying the thermal degradation and combustion as well as the fire retardancy mechanism.
(5) In practice, the fire hazard data obtained can be useful in forest fire management (i.e. it facilitates the selection of the appropriate afforestation species for reducing wildfire danger, for the effective use of fire retardants and the development of new ones, the development of models of fire behavior characteristics in wildland fires, etc.).
(6) In comparison with other flammability test methods [21], the suggested one is sensitive, accurate and reproducible.

It is based on a low cost simple operated apparatus, counting various flammability parameters (i.e. heat content, self-ignition temperature, time to ignition, combustion duration, etc.). The main drawbacks of the method are: (a) the flammability parameters measured are not actual values but relative ones (b) is limited to homogenous particle samples.

Acknowledgements

References

[1] T. Cordero, J.M. Rodrigeuz-Maroto, J. Rodriguez-Mirasol, J.J. Rodriguez, Thermochim. Acta 164 (1990) 135–144.
[2] J. Kaloustian, A.M. Pauli, J. Pastor, J. Therm. Anal. Calorim. 53 (1998) 57–69.
[3] J.M. Rogers, R.A. Sussot, R.G. Kelsey, Can. J. Forest Res. 16 (1986) 721–726.
[4] R.A. Susott, Forest Sci. 28 (1982) 839–851.
[5] S.E. Liodakis, M.K. Statheropoulos, N.E. Tzamtzis, A.A. Pappa, G.K. Parissakis, Thermochim. Acta 278 (1996) 99–108.
[6] A.A. Pappa, N.E. Tzamtzis, M.K. Statheropoulos, S.E. Liodakis, G.K. Parissakis, J. Anal. Appl. Pyrolysis 31 (1995) 85–100.
[7] M. Statheropoulos, S.A. Kyriakou, Anal. Chim. Acta 409 (2000) 203–214.
[8] S. Liodakis, D. Gakis, M. Statheropoulos, N. Tzamtzis, A. Pappa, J. Anal. Appl. Pyrolysis 43 (1997) 139–144.
[9] S. Liodakis, D. Bakirtzis, E. Lois, D. Gakis, Fire Safety J. 37 (2002) 481–494.
[10] S. Liodakis, D. Bakirtzis, A. Dimitrakopoulos, Thermochim. Acta 390 (2002) 83–91.
[11] S. Liodakis, D. Bakirtzis, E. Lois, J. Therm. Anal. Calorim. 69 (2002) 519–528.
[12] S. Liodakis, D. Bakirtzis, A. Dimitrakopoulos, Thermochim. Acta 399 (2003) 31–42.
[13] S. Liodakis, D. Gakis, K. Ahlquvist, M. Statheropoulos, Ann. Forest Sci. 61 (2004) 551–555.
[14] Official Journal of the European Communities 84/449/EEC, No. L251, 19.9.84, pp. 86–88.
[15] H.E. Anderson, Fire Technol. 6 (1970) 312–319.
[16] A.P. Dimitrakopoulos, K.K. Papaioannou, Fire Technol. 37 (2001) 143–152.
[17] A.P. Dimitrakopoulos, Int. J. Wildland Fire 10 (2001) 113–118.
[18] V. Babrauskas, Ignition of wood: a review of the state of the art, Interflam 2001, Interscience Communications Ltd., London, pp. 71–88.
[19] T. Grotjaer, K. Dam-Johansen, A.D. Jensen, P. Glarborg, Fuel 82 (2003) 825–833.
[20] ASTM D 1929–96 (reapproved 2001), Standard Test Method for Determing Ignition Temperature of Plastics.
[21] C.J. Hilado, Flammability Handbook for Plastics, Tochnomic Publishing Co, Inc., Lancaster, 1998, pp 93–98.
[22] C. Lohrer, M. Schmidt, U. Krause, Chem. Ing. Technol. 76 (2004) 936–940.
[23] Y. Chen, S. Mori, W-P. Pan, Thermochim. Acta 275 (1996) 149–158.
[24] S.G. Krigstin, B.M. Wong, D.N. Roy, Wood Sci. Technol. 27 (1993) 309–320.
[25] A. Martli, J. Anal. Appl. Pyrolysis 11 (1987) 149–162.
[26] K.I. Markova, D. Rustschev, Thermochim. Acta 234 (1994) 85–94.
[27] L. Tognotti, A. Malotti, L. Petarca, S. Zanelli, Combust. Sci. Technol. 44 (1985) 15–28.
[28] J. Harris, Thermochim. Acta 14 (1976) 183–199.
[29] C. Huang, T-S. Wu, Thermochim. Acta 239 (1994) 105–114.
[30] E.H.T. Mak, Forest Sci. 34 (1988) 523–529.
[31] J.A. Rodriguez–Anon, F. Fraga-Lopez, J. Proupin-Castineiras, J. Palacio-Ledo, L. Nunez-Regueira, Bioresour. Technol. 52 (1995) 269–274.
[32] L. Núñez-Regueira, J.A. Rodríguez-Añón, J. Proupín-Castiñeiras, A. Vilanova-Diz, Thermochim. Acta 394 (2002) 267–278.
[33] L. Núñez-Regueira, J.A. Rodríguez-Añón, J. Proupín-Castiñeiras, A. Vilanova-Diz, A. Romero-García, Thermochim. Acta 394 (2002) 291–304.
[34] L. Núñez-Regueira, J.A. Rodríguez-Añón, J. Proupín-Castiñeiras, A. Vilanova-Diz, Thermochim. Acta 394 (2002) 279–289.
[35] L. Núñez-Regueira, J.A. Rodriguez-Añón, J. Proupin, A. Romero, Bioresour. Technol. 80 (2003) 121–130.
[36] L. Nunez-Regueira, J. Rodriguez, J. Proupin, B. Mourino, Thermochim. Acta 328 (1999) 105–110.
[37] L. Nunez-Regueira, J. Rodriguez, J. Proupin, B. Mourino, Thermochim. Acta 328 (1999) 111–120.
[38] L. Nunez-Regueira, J. Rodriguez, J. Proupin, A. Vilanova, N. Montero, Thermochim. Acta 371 (2001) 23–31.
[39] L. Nunez-Regueira, J. Rodriguez, J. Proupin, A. Romero, J. Therm. Anal. Calorim. 66 (2001) 281–292.
[40] L. Nunez-Regueira, J. Proupin, J.A. Rodriguez, Bioresour. Technol. 82 (2002) 5–13.
[41] L. Núñez-Regueira, J. Proupín-Castiñeiras, J.A. Rodríguez-Añón, Bioresour. Technol. 91 (2004) 215–221.
[42] A.L. Ulery, R.C. Graham, C. Amrhein, Soil Sci. 156 (1993) 358–364.
[43] A. Wyttenbach, P. Schleppi, L. Tobler, S. Bajo, J. Bucher, Plant Soil 168–169 (1995) 305–312.
[44] ASTM D 4933–91 (reapproved 1997), Standard Guide for Moisture Conditioning of Wood and Wood-Based Materials.
[45] Official Journal of the European Communities 79/831/EEC (reapproved 2003), No. L251, 19.9.84, pp. 86–88.
[46] R.J. Lewis, Rapid Guide to Hazardous Chemicals in the Work Place, second ed., Van Nostrand Reinhold, New York, 1990.