



How to measure the particle ignitability of forest species by TG and LOI

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ABSTRACT

A method was developed to measure the relative particle ignitability (spontaneous and pilot) of forest species using thermogravimetry (TG/DTG) and limiting oxygen index (LOI) techniques. The forest species selected: *Pinus halepensis* Mill., *Quercus coccifera* L., *Pistacia lentiscus* L., *Arbutus unedo* L., *Cistus incanus* L. and forest litter, are dominated in wildland/urban interface (WUI) regions in Mediterranean. The relationships of spontaneous with pilot ignition and ignitability with other flammability parameters (i.e., combustion duration), were investigated. Based on the above ignitability measurements the examined forest species were ranked into categories. Thus, the most ignitable fuel was forest litter and the least one *P. lentiscus*.

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1. Introduction

Wild forest fires comprise a serious problem, burning thousands of hectares all over the world each year. Therefore, the research on flammability of forest species is very important to forest fire management, i.e., facilitates the selection of the appropriate afforestation species for reducing wildfire danger.

In Greece, during the last 30 years, a serious fire problem in wildland/urban interfaces has been observed, mostly intensified around metropolitan and tourist locations. The development of wildland/urban interface (WUI) areas, either due to the expansion of large cities or the development of summer housing, coincides with the increase in both forest fire numbers and burnt areas. The WUI is the area where houses meet or intermingle with undeveloped wildland vegetation and is composed of both interface and intermix communities [1].

Flammability of forest species is a combination of ignitability, sustainability and combustibility. The ignitability determines how easily the fuel ignites. Sustainability is a measure of how well a fire burns with or without a heat source. Combustibility reflects the rapidity with which a fuel is consumed [1,2].

Ignition is the transition between pyrolysis and combustion. It is classified into two types: the pilot and the spontaneous (self-ignition). 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 by heating the fuel.

Valette [3] measured the pilot ignitability of various forest species using an electric radiator, whose radiant surface was a disk of 10 cm in diameter. The method suggested is simple, but very tedious with poor reproducibility. Dimitrakopoulos and Papaioannou [4] have studied the pilot ignitability of foliar lignocellulosic materials using a special constructed ignition apparatus, based on a standard method. Also, the piloted ignition of wood has been determined by a cone calorimeter and a lateral ignition and flame spread test (LIFT) apparatus [5].

However, in the above methods the samples used are whole leaves or stems. The flammability measured with these samples is defined as plant flammability. Plant flammability measurements have poor reproducibility, due to the variation of the involved parameters, such as surface area and packing ratio [6]. On the contrary, using samples after grinding plant into particles eliminates the influence of plant structure. Hence, the particle flammability measurements are consistent and reproducible [1].

The spontaneous particle flammability of solid fuels has been successfully determined by thermal analysis [7–9]. The particle smoldering combustibility of various forest species has been also determined by a specifically designed lab-scale apparatus [10]. The same tests were performed in the presence of fire retardants [7,11].

Limiting oxygen index (LOI) techniques have been widely used for determining the pilot ignitability of polymers, using planar shape samples. Standard LOI tests have been applied to assess the fire resistance of several polymers [12,13] and to evaluate the flame retardant properties of various polymer additives [14–16].

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Nomenclature

CD	combustion duration (min)
DTG	differential thermogravimetry
MWLR	maximum weight loss rate (% °C ⁻¹)
RLOI	relative limiting oxygen index (% v/v)
RSIT	relative spontaneous ignition temperature (°C)
R.S.D.	relative standard deviation
TG	thermogravimetry
WUI	wildland/urban interface

Subscripts

1	refers to gas-phase combustion
2	refers to solid-phase combustion

However, this method has not been yet applied on particle ignitability measurements.

The main objectives of this study were:

- to develop methods for measuring the particle pilot and spontaneous ignitability of forest species by LOI and DTG;
- to find the relationship between pilot and spontaneous particle ignitability for forest species;
- to correlate ignitability with other flammability parameters (i.e., combustion duration);
- to rank forest species into categories, according to their ignitability properties;
- to provide data for developing fire spread models in WUI zones.

2. Experimental

2.1. Samples

Tests were conducted on six forest fuels: *Pinus halepensis* Mill. (Aleppo pine), *Pistacia lentiscus* L. (Mastic tree), *Quercus coccifera* L. (Holly oak), *Cistus incanus* L. (Pink rockrose), *Arbutus unedo* L. (Strawberry tree) and forest litter (leaf litter). The selected species are very common in the Mediterranean region and are often devastated by forest fires. All samples were mature foliage, collected after a long drought period to avoid moisture effects, such as microbes (i.e., fungi, molds and bacteria) growth [17]. The foliage samples were collected from Thrakomacedones, an area at the confines of the national park of mountain Parnitha, northern of the city of Athens. Collection site data of forest samples are shown in Table 1.

According to previous reports [2] the composition of a forest species depends on various factors such as the part of the plant (bark, leaves, etc.), the age of the plant and the climate. Therefore, a special care was taken during the sampling, to collect the most representative gross sample. For each species, a bulk sample comprised of mature leaves detached from three to eight individual plants was collected, in order to minimize interspecies differences [4].

Table 1

Collection site data of forest species

Time of collection	Geographical coordinates	Altitude (m)	Average inclination (%)	Exposition	Dominated petrological formation
25/02/2008 12:00	38°07'50" N 23°46'22" E	323	10	E (97°)	Marls, sandstones, conglomerates
25/02/2008 11:15	38°08'46" N 23°45'26" E	590	60	S (190°)	Limestone, dolomites limestone and dolomite
25/02/2008 10:30	38°08'30" N 23°44'46" E	485	30	S-SW (210°)	Old scree and talus cones

Remarks: the geographical coordinates, the altitude and the exposition were determined by GPS. The sampling area was 50 m around the above-mentioned geographical coordinates and the species selected had a ground cover higher than 5%.

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 vacuum drying procedure was chosen to avoid the vapourization of volatile constituents. The dried samples were ground and a fraction size between 0.1 and 0.2 mm was separated and used for the tests.

The grounded samples were placed into a conditioning box, set at temperature of 32 °C and relative humidity of 12%. The equilibrium moisture content of forest samples was found 2.8%, according to the tables given in a standard method [18]. The tests were performed after reducing plant leaves into a fine uniform substance to eliminate the influence of plant structure and the problems related to heat and mass transfer [19]. Thus, the results taken are more consistent, counting the intrinsic components (i.e., chemical composition) of forest species, suitable for comparison use.

The samples for the LOI method should be self-supporting or thin films, according to ASTM D2863-00. Thus, we prepared pellets as follows: 0.50 g ground forest species of 0.1–0.2 mm were pressed into cylindrical pellets of diameter of 19 mm and height of 1.5 mm, at a pressure of 276.8 MPa in order to produce uniform samples. Then, the samples were stored in a desiccator, using as drying agent silica gel; hence, the moisture absorbance was avoided, whereas the recession of temperature to 25 °C, necessary for the LOI test, was succeeded.

2.3. Relative limiting oxygen index (RLOI)

Limiting oxygen index measurements were carried out using a Dynisco Limiting Oxygen Index Chamber. A method has been developed to determine the particle pilot ignitability of forest species, based on a standard method [20]. According to the method suggested, LOI measurements were conducted at ambient temperature, with a 20-mL s⁻¹ gas flow of oxidative media passing through a quartz glass column of 95 mm diameter. The forest species pellets, were placed vertically in the center of the glass column, using a sample holder. The oxygen ratio variation in the gas flow was 0.2%, whereas the lowest visible part of natural gas flame was applied to the top edge of pellets for 10 s. Relative limiting oxygen index was defined as the lowest oxygen concentration (% v/v) in the carrier gas flow at which full flaming combustion of the pellets was observed. RLOI is a measure of pilot ignitability; thus, highly ignitable fuels have low RLOI values.

2.4. Thermogravimetric (TG/DTG) analysis

Thermogravimetry (TG) and differential thermogravimetry (DTG) were carried out on a Mettler Toledo TGA/SDTA 851 apparatus, using samples of 20 mg and an open type alumina (Al₂O₃) sample holder. The samples were heated under non-isothermal conditions (25–600 °C) with a linear heating rate of 10 °C min⁻¹.

All runs were conducted in air atmosphere with a flow rate of $100 \text{ mL} \cdot \text{min}^{-1}$, to ensure complete combustion. The particle flammability measurements of forest species were based on the following DTG analysis data, recorded by the STAR-e software system of Mettler Toledo TG/SDTA 851 apparatus:

1. Onset of gas-phase combustion, in $^{\circ}\text{C}$, defined as the relative spontaneous ignition temperature (RSIT).
2. DTG peak height, in $\% \cdot \text{C}^{-1}$, which is the maximum weight loss rate (MWLR) during combustion, and is related to the combustion rate.
3. Endset minus onset of DTG peaks, in min, defined as the combustion duration (CD).

MWLR₁ and CD₁ refer to the gas-phase combustion, whereas MWLR₂ and CD₂ to the solid-phase one.

3. Results

3.1. Relative LOI

The relative oxygen index values of the forest fuels examined are presented in Fig. 1. The data given in Fig. 1 are the mean values of three replicate measurements, with R.S.D. lower than 1%.

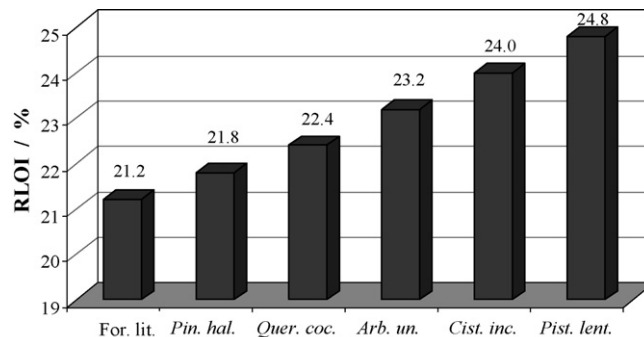


Fig. 1. Relative limiting oxygen index values of WUI forest species: data were arranged from low to high values.

According to the RLOI data given in Fig. 1, forest litter is the most pilot ignitable fuel, whereas *P. lentiscus* is the least one.

3.2. Thermogravimetric analysis

The TG and DTG curves of the forest species analysed are shown in Fig. 2. Under the thermal analysis combustion (air atmosphere) conditions used, two exothermic peaks (DTG) were observed. The

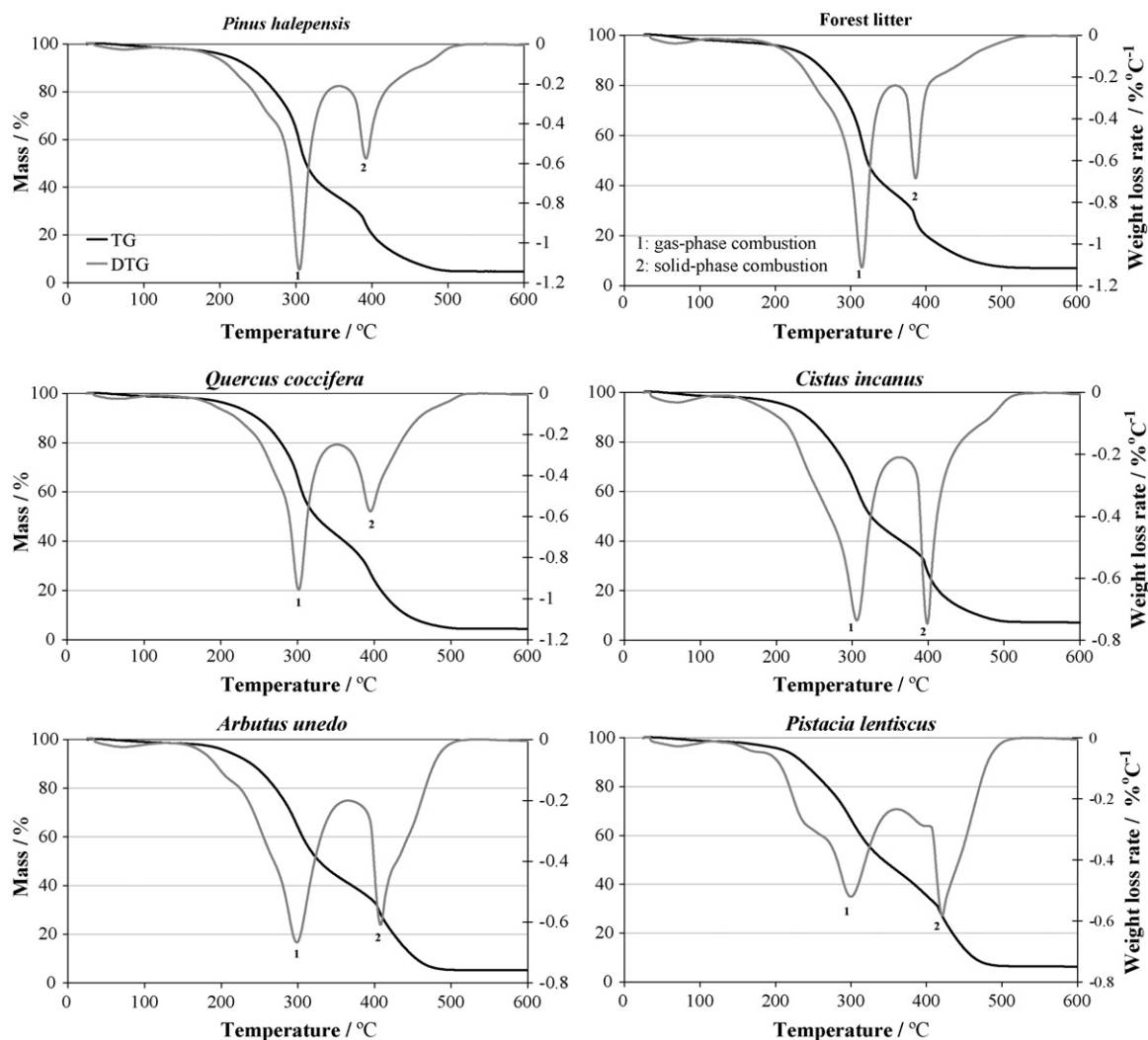


Fig. 2. TG and DTG curves of WUI forest species in air atmosphere.

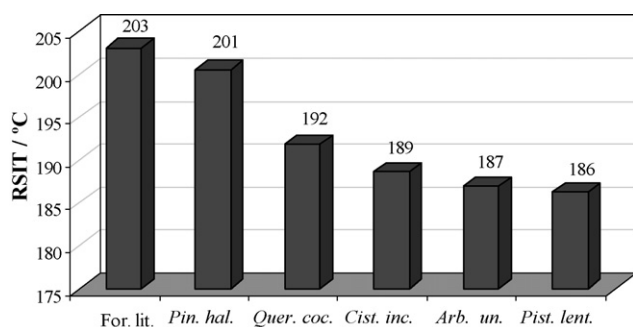


Fig. 3. Particle relative spontaneous ignition temperature (RSIT) values of WUI forest species: data were arranged from high to low values.

first (around 310 °C), is attributed to the gas-phase combustion, whereas the second (around 410 °C), is attributed to the solid-phase combustion of char, known as smoldering combustion [1,8,21].

The relative spontaneous ignition temperatures of forest species, determined by the onset of first DTG peaks (Fig. 2) are presented in Fig. 3. The data given are the mean values of three replicate measurements, with R.S.D. lower than 3%.

According to previous reports, the spontaneous ignitability is related to the pilot one [2,22]. Thus, fuels with high spontaneous ignition temperature (or ignition delay time) have low pilot ignition temperature (or flash point values); therefore, burn easily by flame and are characterized as ignitable fuels. On the basis of the above statement, the most ignitable fuels are, forest litter and *P. halepensis* and the least ignitable one, is *P. lentiscus* (Fig. 3).

The maximum weight loss rate of gas-phase combustion ($MWLR_1$), expressed in % °C⁻¹, is related to the combustibility: combustibility increases as $MWLR_1$ value increases [1,19,21]. Thus, *P. lentiscus* is the least combustible fuel and *P. halepensis* is the most combustible one.

The gas-phase combustion duration is also related to the combustibility: the most combustible fuels have shorter combustion duration values [1]. Thus, forest litter and *P. halepensis* are the most combustible fuels, whereas *P. lentiscus* is the least combustible one.

4. Discussion

A linear relationship was found between pilot (RLOI) and spontaneous ignitability (RSIT), with R^2 value of 0.81, as shown in Fig. 4. This inverse proportional relationship is in agreement with previous reports: the most ignitable fuels have high spontaneous and low pilot ignition temperature [2,22]. Thus, the most ignitable forest species are those placed on the right bottom side of the diagram (i.e., forest litter, *P. halepensis*), whereas the least ignitable ones are those placed on the left top side of the diagram (i.e., *P. lentiscus*).

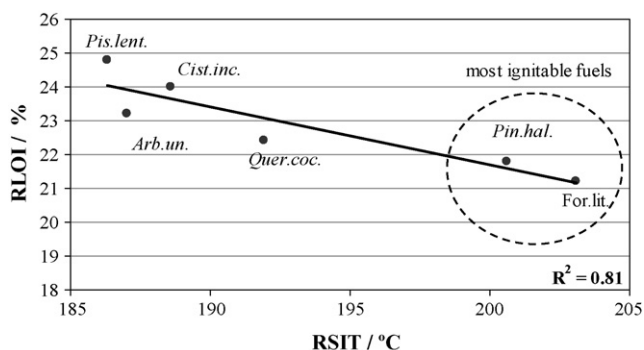


Fig. 4. Correlation between pilot (RLOI) and spontaneous ignition (RSIT).

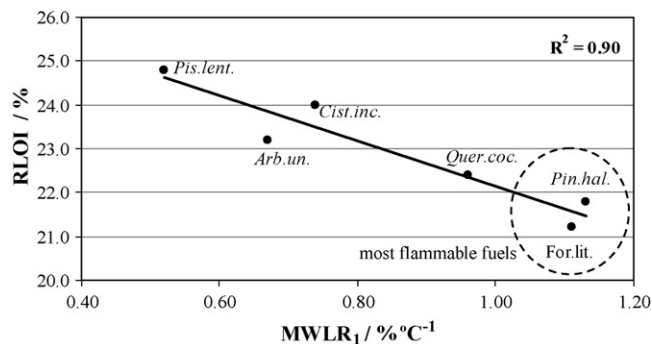


Fig. 5. Correlation between pilot ignition (RLOI) and maximum weight loss rate of gas-phase combustion ($MWLR_1$).

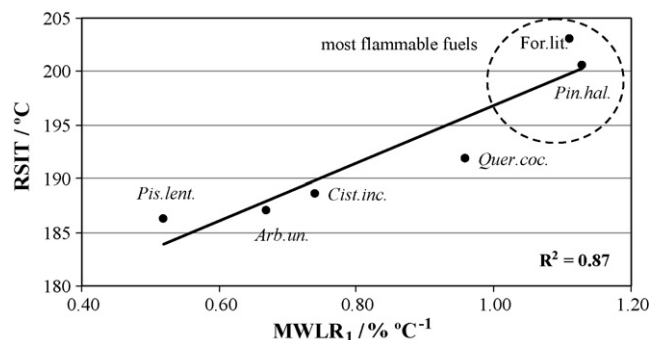


Fig. 6. Correlation between spontaneous ignition (RSIT) and maximum weight loss rate of gas-phase combustion ($MWLR_1$).

Linear relationships were also found between the ignitabilities (pilot and spontaneous) and the maximum weight loss rate of gas-phase combustion, as shown in Figs. 5 and 6. Therefore, ignitability is related to the rate of gas-phase combustion, with the most ignitable fuels having the highest gas combustion rates. On the basis of Fig. 5, the forest species examined can be ranked, according to their flammability properties. Thus, the most flammable fuels in terms of ignitability and gas-phase combustibility are located on the right bottom side of Fig. 5 (i.e., forest litter and *P. halepensis*), whereas the least flammable are those located on the left top side (i.e., *P. lentiscus*).

Similarly, the ignitabilities (pilot and spontaneous) of forest species examined are linearly related to the combustion duration of gas-phase combustion (CD_1), as shown in Figs. 7 and 8. Hence, the proportional linear relationship between ignitability and gas-phase combustibility of forest species was confirmed. Also, the forest species ranking, according to their flammability properties

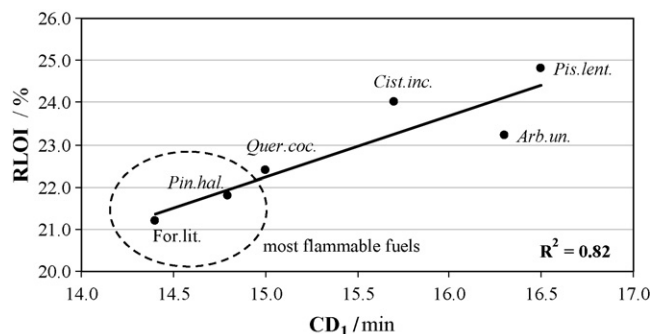


Fig. 7. Correlation between pilot ignition (RLOI) and gas-phase combustion duration (CD_1).

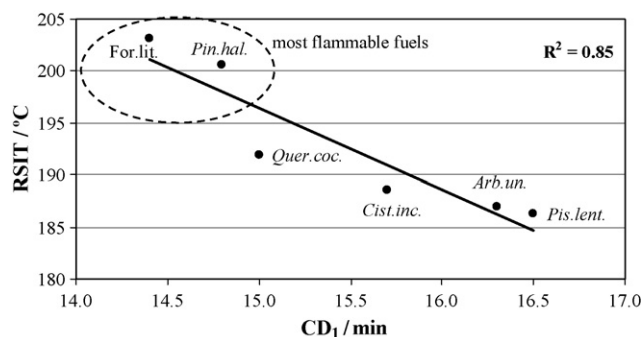


Fig. 8. Correlation between spontaneous ignition (RSIT) and gas-phase combustion duration (CD₁).

previously reported, was confirmed: the most flammable fuels in terms of ignitability and gas-phase combustibility are located on the left bottom side of Fig. 7, whereas the least flammable ones are on the right top side.

On the contrary, no obvious relationship could be established between ignitability and solid-phase maximum weight loss rate (MWLR₂) or combustion duration (CD₂). It seems that solid-phase (char) combustion is not related to the ignition. Combustion of char usually follows gas-phase combustion, whereas ignition initiates gas-phase combustion [23]. On this matter, further investigation is required. Other lab-scale methods have been developed to determine spontaneous ignition temperature of solid-phase combustion [2,24].

5. Conclusions

The main conclusions drawn out of this work are as follows:

- Two simple methods were developed for measuring the relative particle ignitability of forest species. The first concerns pilot ignitability and is based on LOI technique. The second concerns spontaneous ignitability and is based on DTG analysis.
- An inverse linear relationship is established between pilot (RLOI) and spontaneous ignition (RSIT) data for forest species.
- The ignitability is linear related to the gas-phase combustibility of forest species. On the contrary, no correlations between ignitability and solid-phase combustion are established.
- On the basis of data provided, the WUI forest species examined were ranked, according to their flammability properties.
- The ignition and combustion data provided are relative ones and are useful only for comparison reasons.
- The influence of sample status on flammability properties requires further investigation. However, the use of different sam-

ples (powder/pellets) required by the experimental methods developed, does not degrade the correlations extracted, since the data provided are relative and are only for comparison reasons.

- The ungrounded samples are not expected to exhibit the same ignitability properties as the grounded ones. According to previous studies, the flammability depends on the surface area, the volume and the particle density of forest leaves [25]. Therefore, further work is suggested for measuring plant flammability and to compare with the particle one.

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