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Thermochimica Acta 390 (2002) 83–91

thermochimica acta

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Ignition characteristics of forest species in relation to thermal analysis data

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Received 5 February 2002; accepted 11 February 2002

Abstract

The ignitability of various forest species was measured with a specifically designed apparatus, under precisely controlled temperature and airflow conditions. The ignitability tests were based on ignition delay time versus temperature measurements using five different forest species: *Pinus halepensis*, *Pistacia lentiscus*, *Cupressus sempervirens*, *Olea europaea*, *Cistus incanus*. These species are common in the Mediterranean region and frequently devastated by forest fires. The ignition characteristics of the forest fuels examined were related to thermogravimetric analysis data. The DTG curves showed that the mass changes related to cellulose decomposition in the temperature range of 320–370 °C are greatly responsible for the ignition behavior of the species tested. In addition, the mass of volatiles evolving between 120–160 °C has a significant effect on the ignitability. On the contrary, the inorganic ash content of forest fuels, measured by atomic absorption spectroscopy, seems to play an insignificant role on the ignitability characteristics of the forest fuels examined. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Forest fuels; Ignition test; Thermogravimetric analysis

1. Introduction

Forest fuel burning during wildland fires initially involves a series of endothermic reactions known as *preignition*. During this phase and as the fuel is heated, dehydration and volatilization of organic substances occur. Volatile compounds such as terpenoid hydrocarbons volatilize at low temperatures, while at higher temperatures begins the process of *pyrolysis* of the forest fuels. Pyrolysis breaks down the substance molecules into low molecular mass gases (volatiles), tars, carbonaceous char and mineral ash. Cellulose, which is a condensation polymer of the hexose sugar D-glucose, is a principal component of forest species, comprising 41–53% w/w of the total weight. The thermal degradation of cellulose takes place between 280–400 °C and follows two competing pathways [1,2]. One is the dehydration, which leads to char, and gases (mainly CO, CO_2, H_2O) and the other is the depolymerisation, which leads to tar and volatiles through the formation of levoglucosan. High temperatures and high heating rates favor the evolution of volatiles which mainly support combustion, while low

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PII: S0040-6031(02)00077-1

temperatures and slow heating rates promote the production of tar and char. Hemicelluloses comprise 15–25% w/w of the forest species, have similar chemical structure to cellulose, based on pentose and hexose sugars and therefore, exhibit similar thermal degradation behavior. Lignin (16–33% w/w) is a more complex and thermally stable component of forest species. It is pyrolysed in the temperature range of 280–500 °C, contributing more to the char formation [2,3].

The second phase in the burning process is exothermic and it is known as *combustion*. Combustion may be with or without flame. *Flaming combustion* is a gas oxidation phase, accompanied by emission of flames. It takes place when the temperature of volatiles, emerging through the surface of the forest fuels, reaches 450–500 °C [2].

The combustion of a solid without flames is termed *smoldering combustion* [4]. Smoldering is associated with surface oxidation of the char, which provides the heat necessary to cause further pyrolysis of the neighboring layer of combustible material. In the smoldering combustion mode we may distinct three regions [5]: the pyrolysis zone in which there is a steep temperature rise and an outflow of visible volatile products (smoke); the charred zone where the temperature reaches its maximum value, the evolution of the visible products ceases and glowing occurs; the porous residual char/ash zone, which is not glowing and whose temperature is decreasing slowly.

Smoldering combustion is an important part of forest fires because it is related to ground fires in the organic soil horizons which could last for months or even years. In general high temperatures favor the evolution of volatiles flammable gases, whereas low temperatures promote the production of tar and char leading to smoldering combustion. Smoldering generally occurs in fuel arrays that are more tightly packed than those that sustain flaming combustion [4].

Ignition is the transition between the preignition and combustion phase, the temperature at which a pilot source of heat is no longer required. Once ignited, the heat generated by the combustion brings other fuel to ignition temperature, thus spreading the fire.

Wood is considered as one of the most common combustible materials and there have been numerous studies relating to its ignition. There is a number of reviews concerning the ignition of wood [6,7]. Atteya performed a comprehensive set of ignition measurements on eight wood species [8]. Kanuary has found that the minimum surface temperature (critical surface temperature) of wood under radiative heating mode for spontaneous ignition is 600 °C and for piloted ignition 300–410 °C. With convective heating the spontaneous ignition, occurred at 490 °C and with piloted ignition 450 °C [7]. In addition, 2.5 g m^{-2} s^{-1} is reported as the minimum supply of fuel vapors (pyrolysis rate) for ignition. From the literature is shown that both the minimum pyrolysis rate and the minimum surface temperature can be used as criteria for ignition. However, from a practical point of view, the surface temperature criterion is much more convenient. Núñez-Regueira et al. [9–11] had determined the calorific values and flammability of various forest species in relation to their elementary chemical composition, moisture, density and ash percentage after combustion. Based on this work, the above researchers designed risk index maps [12–16] used as a tool to prevent forest wildfires.

Thermal analysis techniques can be used for measuring the volatilization rate of forest fuels as a function of temperature [17]. Rogers et al. [18] related the TGA thermal behavior of a forest material with its chemical composition. Also, Kaloustian et al. [19] using DTA and TGA correlated the maximum thermal decomposition rate of various Mediterranean plants with their content in cellulose. Cordero et al. [20] used TGA to study the kinetics of thermal decomposition of *Eucalyptus sawdust*. Other researchers have examined the effects of various fire retardants on the pyrolysis of forest fuels using TGA and DTA [21–24]. However, the small 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 ignitability performance in field conditions. Therefore, the information provided by analytical methods should be supported by large-scale tests, approaching the actual wildland fire conditions.

In this paper, the spontaneous ignition characteristics of five forest fuels common in Mediterranean region, has been studied using a specifically designed apparatus, under precisely controlled temperature and airflow conditions. In addition, the forest species have

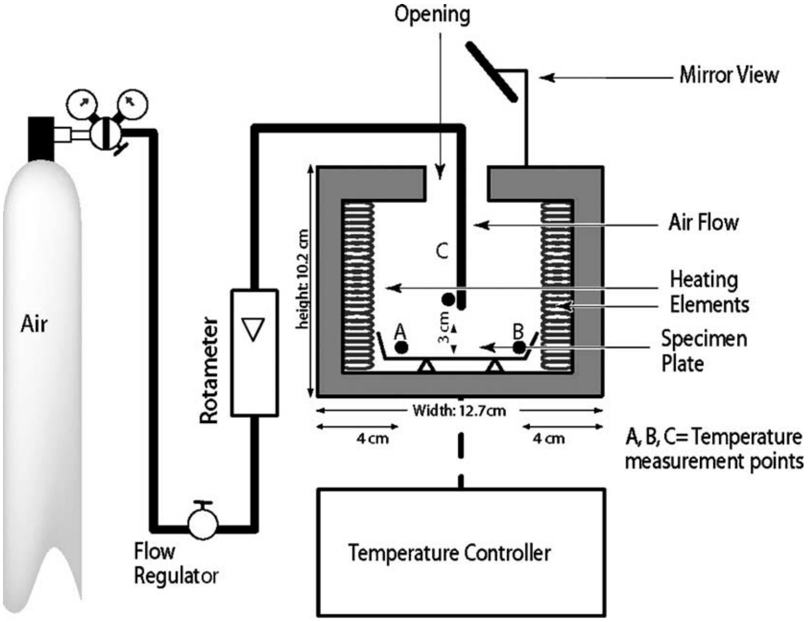

Fig. 1. Diagrammatic presentation of the apparatus used for measuring the spontaneous ignitability of forest species.

being analyzed by TGA and their inorganic constituents were determined by atomic absorption spectroscopy. The main purpose of this study is to interpret the ignitability characteristics of forest fuels using thermal analysis methods.

2. Methodology

2.1. Apparatus for ignition measurements

The apparatus used for the ignition tests is presented in Fig. 1. The furnace has internal dimensions 12.7 cm × 10.2 cm × 15.2 cm (width, height, depth) and the temperature control accuracy was ±1 °C from ambient to 1200 °C, whereas the temperature uniformity was ±3 °C at 1000 °C. The forest samples, in the form of cylindrical pellets of 1 g, were dropped into a porcelain plate of 8 cm diameter, located at the bottom of the oven chamber, through a circular opening from the top of the furnace, as shown in Fig. 1. Three Chromel/Alumel thermocouples 0.05 mm diameter, were used to monitor the oven temperature with an accuracy higher than 0.3 °C. Thermocouples T_1 and T_2 were located at points A and B, respectively, to monitor the surface temperature of the porcelain plate, while thermocouple T_3 was located 3 cm above the plate, approximately at the geometric center of the oven (see Fig. 1). The oven opening was also used to observe via a mirror any changes in the specimens (ignition, smoking, smoldering) as well as for ventilating the system (removal of combustion gases). A controlled flow of air in the range of 400 to 3,000 ml min^{-1} ±10% was admitted at the bottom of the oven, measured by a rotameter. The air entered the chamber through a circular stainless steel pipe jet, whereas the airflow was adjusted through an accurate flow regulator. Also, the incoming air was at room temperature, without causing any substantial change on temperature settings.

2.2. Instruments for the analysis measurements

Atomic Absorption Spectrometer (Perkin Elmer instrument, model 3300) was used for the determination of inorganic constituents of forest species.

Thermal analyses were carried out on a Mettler TGA/SDTA 851 apparatus. A linear heating rate of 10 °C min^{-1} was applied for all thermal tests. The carrier gas used was nitrogen and the flow rate was

Table 1
Description of forest samples tested (density of ground forest species within 0.3–0.5 nm, heat of combustion at 25 °C)

Material	Density (g ml^{-1})	Heat of combustion (kJ kg^{-1})
P. halepensis	0.45	22.185
P. lentiscus	0.51	22.018
C. sempervirens	0.37	19.548
O. europaea	0.42	21.373
C. incanus	0.42	21.683

Sampling data: The location that the samples were collected was the mountainside Kalopoula of Imitos, near Athens, located W-NW with geographic coordinates 37°58′13″N and 23°48′20″E, height 487 m and average slope 70%. The mature foliage samples, taken from stems 4–6 years old, were collected after a long dry period in order to avoid making moldy. The soil was limestone and the dates of collection were 15 March and 7 May 2000.

adjusted at 50 ml min^{-1}. The sample size was about 10 mg, whereas the aluminum sample holder was open.

2.3. Samples

Tests were conducted on five forest fuels; *Pinus halepensis*, *Pistacia lentiscus*, *Cupressus sempervirens*, *Olea europaea*, *Cistus incanus*. The selected forest species are common in the Mediterranean region and are often devastated by forest fires. Some characteristics of the forest materials tested are shown in Table 1.

The heat of combustion of the forest samples was determined by a Parr Instruments Company plain jacket bomb calorimeter, model 1341. The density of the pellets was approximately 1.2 g ml^{-1}.

2.3.1. Sample preparation for the ignition measurements

The samples were prepared according to the following procedure: the samples were dried at 60 °C, under vacuum of 100 mmHg until constant weight within 1 mg. The approximate time of drying the samples was 48 h. The dried samples were then ground and a fraction between 0.3 and 0.5 mm was separated and used for the tests. For the ignition measurements about 1 g of the powder was pressed into a cylindrical pellet of diameter of 1.3 cm at a pressure of 15.7 MPa (155 atm) in order to produce uniform samples. The pellets were dried (60 °C and 100 mmHg vacuum) and then stored in a desiccator, using as drying agent silica gel, until the ignition test.

2.3.2. Sample preparation for analysis measurements

For the inorganic constituent measurements the forest species after drying and grinding were heated in an open porcelain crucible until all carbonaceous matter had been oxidized, leaving a residue of inorganic components (dry ashing). This residue was then dissolved in dilute HCl to give a solution, which can be analyzed by atomic absorption spectroscopy. The samples for the thermal analysis were prepared with the drying and grinding procedure described in Section 2.3.1.

2.4. Procedure

The temperature of the unloaded furnace was maintained for at least 15 min to assure stability, whereas the regulation of the current through the heating coils was not changed during the test. The forest fuel samples were dropped into the furnace vessel through the circular opening from the top of the furnace and the time of any change of the specimens (flaming, smoking, smoldering) was recorded.

After each test, the porcelain vessel was taken out of the chamber for cleaning. Sufficient time was given, after placing back the vessel, to allow the setting of steady conditions inside the chamber.

3. Results and discussion

The ignition delay time of the forest species tested, in the temperature range of 500–600 °C and airflow of 1.5 l min^{-1}, is presented in Table 2.

The measurements reported in Table 2 are the average of five replicate tests, while the relative standard deviations determined were below 10.

From the data shown in Table 2, the trendlines of ignition delay time versus temperature, for every species, were plotted as presented in Fig. 2. Based on the above graph the forest fuels examined can be classified from the least to the most flammable species as follows: *P. lentiscus*, *C. incanus*, *O. europaea*, *C. sempervirens*, *P. halepensis*.

The major inorganic constituents of forest species were determined by atomic absorption spectroscopy after dry ashing the samples at 600 °C, according to

Table 2
Ignitability properties (ignition delay at various temperatures) of forest fuels, using samples of 1.0 g and airflow of 1.5 l min^{-1}

Temperature (°C)	Ignition delay (s)				
	Forest species				
	P. halepensis	*P. lentiscus*	*C. sempervirens*	*O. europaea*	*C. incanus*
460	NI	NI	NI	NI	NI
470	NI	NI	NI	NI	NI
480	45	NI	55	57	NI
490	35	NI	43	56	61
500	30	NI	36	42	48
510	30	NI	36	35	43
520	24	33	27	27	37
530	21	35	24	23	31
540	14	23	18	22	29
550	14	18	12	17	27
560	14	18	15	15	21
570	12	17	13	10	20
580	10	12	11	7.5	17
590	10	9.0	10	9.0	12
600	9.5	9.0	7.5	7.0	11

NI: no ignition.

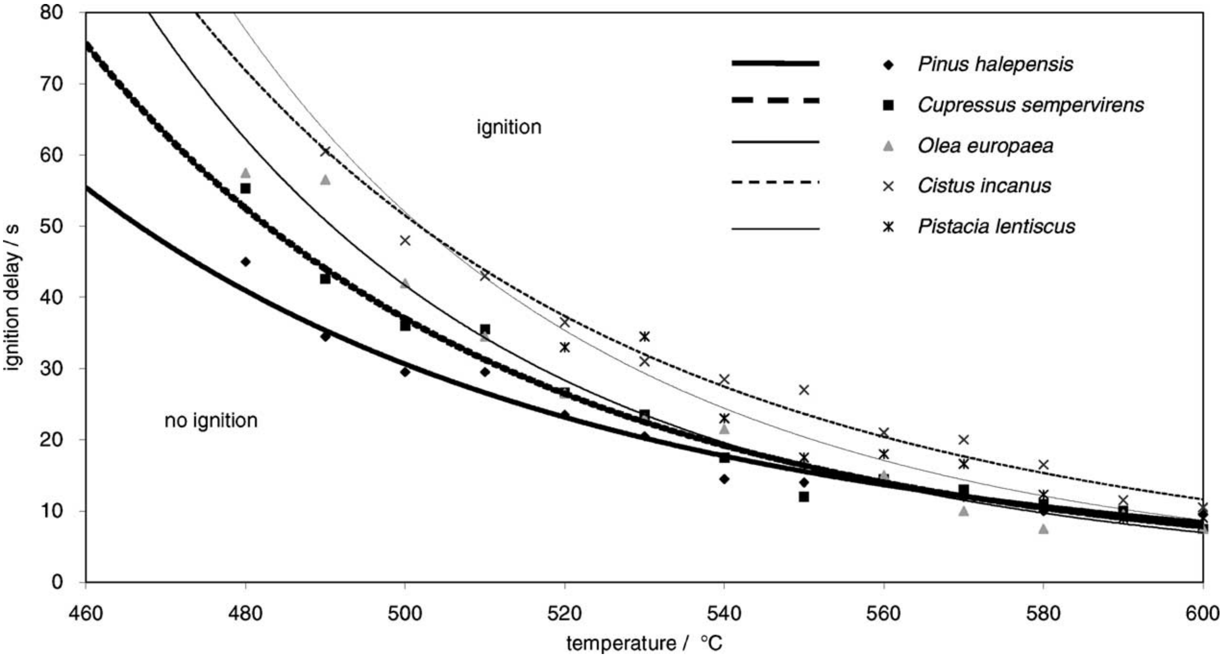

Fig. 2. Ignitability behavior of various forest species, under constant sample size of 1.0 g and constant airflow of 1.5 l min^{-1}.

Table 3
Ash and major inorganic constituents content of forest species tested in relation to ignition data

Forest species	Ignition delay at 500 °C (s)	Ash content (% w/w)	Inorganic constituents (% w/w)				
			Ca	Mg	K	Na	Fe
P. lentiscus	NI	4.41	8.87	2.14	8.87	0.39	0.13
C. incanus	48	5.06	11.5	2.56	4.44	0.30	0.37
O. europaea	47	5.64	32.3	2.60	7.64	2.87	0.20
C. sempervirens	36	3.52	21.0	2.25	5.76	0.25	0.41
P. halepensis	30	3.51	16.6	2.08	4.58	0.44	0.22

standard methods [25]. It is reported that in most cases, alkali and alkaline earth elements such as Ca, Mg and K make up about 80% of the total inorganic constituents of wood [26]. The ash content with its major elemental composition of the forest fuels examined is presented in Table 3.

The measurements reported in Table 3 are the average of four replicate tests, while the relative standard deviations determined were below 10.

It has been reported that in plant materials with higher ash contents, maximum volatilization rates decrease, residues increase and pyrolysis begins at lower temperatures [3]. This is expected to reduce the ignitability of the samples.

The data shown in Table 3 do not clearly confirm this relationship between ash content and the ignitability for the forest species examined. In addition none of the inorganic constituents determined seems to have a direct effect on the ignitability behavior. Nevertheless, the above experimental data could be used to classify the forest species into two groups: the first includes the least flammable species which have ash contents between 4.41–5.64% w/w and the second the most flammable ones with an ash content between 2.96–3.52% w/w.

The TGA and DTG curves of the forest species examined in a nitrogen atmosphere are shown in Figs. 3–7. All TGA graphs have similar profiles. Based on the TGA data, the mass residues at 600 °C were determined. These masses decrease as the ignitability increases (ignition delay decreases), as is shown in Table 4. Thus, the ignitability, as expected, is related to the total mass evolved during the pyrolysis stage.

In the DTG graphs (mass change with respect to temperature, d*m*/d*T* versus temperature *T*), the overlapping reactions, i.e. thermal decomposition of cellulose, hemicelluloses and lignin, are partially resolved. In addition, the profile of the DTG curves

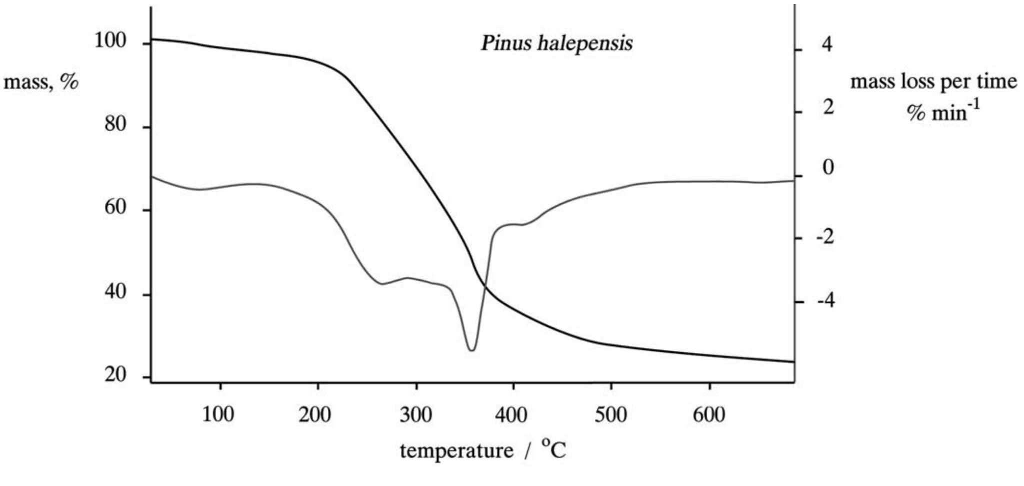

Fig. 3. TGA and DTG curves of *P. halepensis* in N_2 atmosphere.

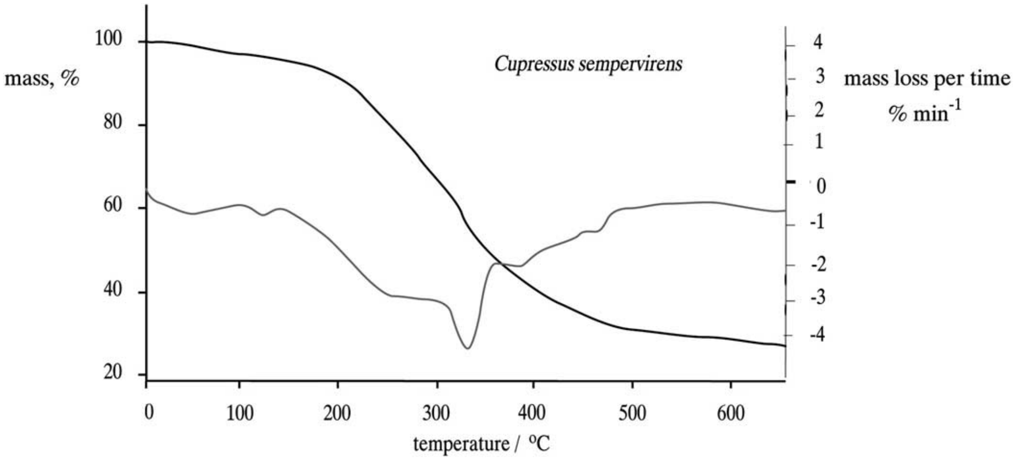

Fig. 4. TGA and DTG curves of *C. sempervirens* in N_2 atmosphere.

varies depending on the forest species examined. Based on the DTG graphs it is observed that appreciable weight losses take place in five different temperature ranges. The first, in the range of 30–100 °C, corresponds mainly to the drying process due to the evaporation of moisture. The second, in the range of 120–160 °C, appeared only in DTG diagrams of *O. europaea* and *C. sempervirens*, and is attributed to the evaporation of volatile constituents. The peak in the range of 200–280 °C corresponds mainly to the hemicellulose decomposition, while the next one between 320–370 °C is related to the cellulose decomposition [1,2,24]. The final is the most pronounced peak in the DTG diagrams. The last peaks in the range of 370–500 °C are related to the lignin decomposition [1,2,24].

Correlating the ignition properties with the DTG analysis data (see Table 4) it is shown that the ignitability mainly depends on the amount of cellulose decomposed during pyrolysis at 320–370 °C. This means that the ignition delay time increases as the amount of cellulose decomposed to inflammable gases decreases. Another important factor, which determines the ignition properties of forest fuels, is the amount of volatiles evolving in the temperature range

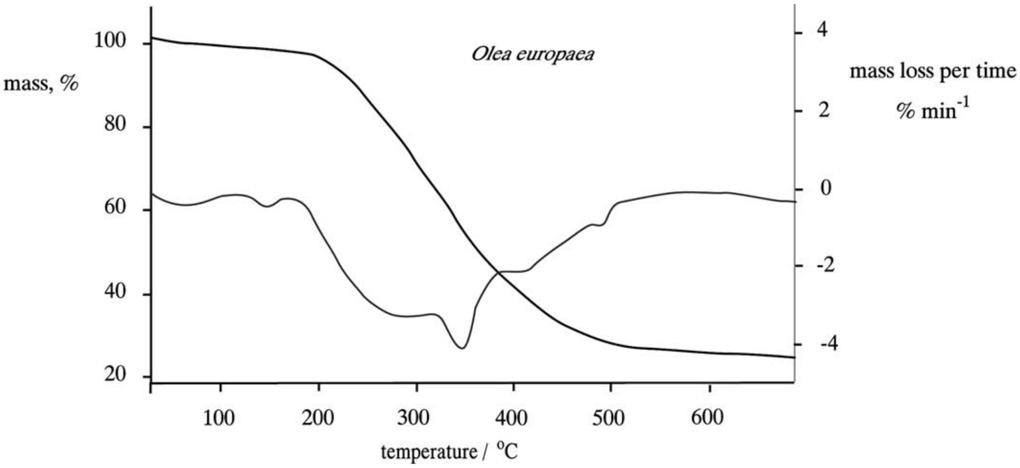

Fig. 5. TGA and DTG curves of *O. europaea* in N_2 atmosphere.

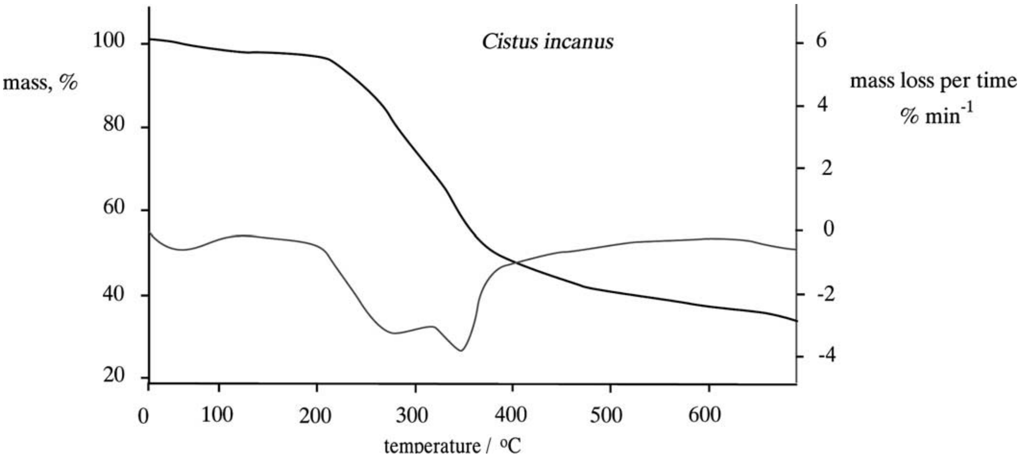

Fig. 6. TGA and DTG curves of *C. incanus* in N_2 atmosphere.

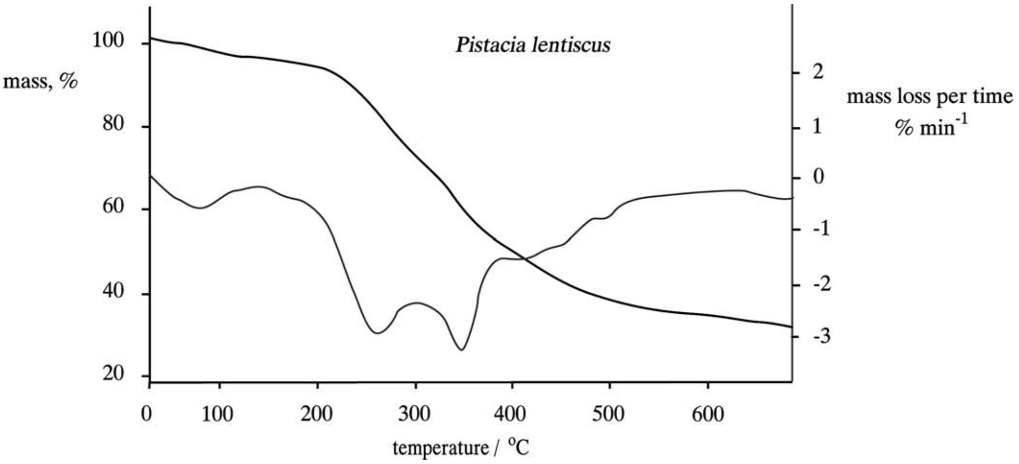

Fig. 7. TGA and DTG curves of *P. lentiscus* in N_2 atmosphere.

Table 4
Mass changes based on TGA analysis in relation to ignition data for various forest species

Forest species	Ignition delay at 500 °C (s)	Mass residue at 600 °C (% w/w)	Relative mass changes (% w/w) in the temperature range (°C)				
			35–100	120–160	200–280	320–370	370–500
P. lentiscus	NI	34.4	2.23	0.83	16.0	13.8	16.9
C. incanus	48	37.1	1.47	0.54	15.1	16.6	11.6
O. europaea	47	25.5	1.38	0.92	18.5	17.1	20.7
C. sempervirens	36	28.9	2.07	1.48	14.3	16.8	19.8
P. halepensis	30	23.2	1.31	1.10	18.7	21.1	16.0

120–160 °C. This probably explains the relative high flammability of *C. sempervirens*.

4. Conclusions

The ignition properties of forest species determined at laboratory scale may be interpreted by DTG analysis data. First the ignitability mainly depends on the amount of cellulose decomposed during pyrolysis between 320–370 °C, i.e. the ignition delay increases as the amount of cellulose thermally decomposed increases. Second, the ignitability increases as the total mass residue after thermal treatment up to 600 °C decreases. Also, the mass of volatiles evolved between 120–160 °C play an important role on the ignition. On the contrary, the inorganic constituents content seems to have a minor influence on the ignition properties of forest fuels determined by AAS. Consequentially, thermal analysis data may facilitate to a better understanding of the mechanism of ignitability and furthermore on fire retardation studies.

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