ACTA FACULTATIS XYLOLOGIAE ZVOLEN, 59(2): 79−90, 2017
Zvolen, Technická univerzita vo Zvolene
DOI: 10.17423/afx.2017.59.2.08

COMPARISON OF THE ACTIVATION ENERGY REQUIRED FOR SPONTANEOUS IGNITION AND FLASH POINT OF THE NORWAY SPRUCE WOOD AND THERMOWOOD SPECIMENS

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ABSTRACT

The important fire characteristics include also the data related to the material activation energy, which determination enhances the understanding of the combustion process, reasons of fire initiation and the typical behaviour of specific materials in their thermal degradation process. They can be also used as input parameters when modelling the dynamics of fires, on which base can be assessed existing fire safety measures and further proposed new one, more efficient, encompassing also the suitable design of building construction, especially of wooden buildings. Except the sophisticated procedures to determine the activation energy of materials, using e.g. the cone calorimeter, there are still in use the mathematical models, which often represent simplified procedures to determine them without any need to have such an expensive equipment. In the paper, there are described the particular procedures to determine the activation energy of the spontaneous ignition / flash point of the Norway spruce wood and Norway spruce Thermowood specimens tested. The specimens were represented by the prisms with dimensions of 20 × 20 × 10 mm. The calculation of the spontaneous ignition / flash point activation energy of the two types of specimens tested was carried out in accordance to the analogy of the equation of Arrhenius for calculating the activation energy. Spontaneous ignition / flash ignition induction periods of the specimens, tested at various temperatures, were determined by the Setchkin Furnace test, in accordance to the STN ISO 871 standard. Finally, the resulting values of the spontaneous ignition / flash point activation energy for Norway spruce wood and Thermowood were compared. There was found that higher spontaneous ignition / flash point activation energy was necessary to be added to Norway spruce wood specimens and lower one to the Thermowood specimens. However, the differences in the activation energy values are not so much significant as expected.

Key words: Arrhenius`s equation, activation energy, induction period, Norway spruce wood, Norway spruce Thermowood.

INTRODUCTION

In terms of a comprehensive assessment of all the factors affecting the possibility of fire initiation, it is necessary to know especially the fire ignition properties of materials, which are the quantitative expressions of the possibility of fire initiation. They represent the response of materials to a high temperature or fire. For better usability of the fire ignition parameters, it is appropriate a parallel monitoring the conditions affecting the course of the combustion process initiation period, i.e. the fire tetrahedron (CHREBET, BALOG 2010 in terms of NFPA 921: 2008).

Thermal conditions for initiating the burning process are usually characterized by the flash point and ignition temperature (the ignition parameters). Their determination is stated in the STN ISO 871. In the assessment of ignition sources and combustible transfer, there are used the technical and safety parameters like spontaneous and flash point temperatures.

Determination of spontaneous ignition / flash point temperature is closely linked to other technical safety parameter - the induction period. The induction period, together with the temperature, characterizes processes related to materials before flame occurrence, thus with increasing reaction temperature it is reduced. In general, we distinguish between the spontaneous ignition induction period, i.e. the time interval from the specimen thermal loading up to its spontaneous ignition, without the use of a small flame as the secondary source of ignition, while its time interval is equal to or shorter than 10 minutes and flash point induction period, the time interval from the specimen thermal loading, but using a small flame as the secondary ignition source (BABRAUSKAS 2003).

The spontaneous ignition parameters, together with the induction spontaneous ignition and flash point temperature are essential for the relative comparison of materials in terms of the fire dynamics in their thermal loading by temperature or heat flux, which are typical for a fire, i.e. they characterize the initiation of combustion (BABRAUSKAS 2003).

One of the variables describing the behaviour of wood in a fire is the activation energy required for the spontaneous ignition / flash point. That is, how much energy (heat) is necessary to provide a specific form of wood to be spontaneously ignited / flash ignited during the thermal loading. The range of activation energies for spontaneous ignition / flash point of wood is quite wide and default values depend significantly on the methodology applied (RANTUCH *et al.* 2016).

Ignition parameters as well as activation energy setting, in general, is closely related to the issue of combustion kinetics evaluation. Here we introduced several studies focusing the Norway spruce wood.

The influence of thermal loading at spruce wood lignin alteration, which has also impact on the change of combustion kinetics, studied KAČÍKOVÁ, KAČÍK (2009).

KUČEROVÁ *et al.* (2011) further published a study dealing with alterations of extractives and cellulose macromolecular characteristics after thermal degradation of Norway spruce wood.

The influence of spruce wood heat treatment on its thermal stability and burning process has studied MARTINKA *et al.* (2014).

MARTINKA *et al.* (2015a) further dealt with the evaluation of the Norway spruce wood form influence on its activation energy of spontaneous ignition. They investigated the dust, solid and pellet forms of the specimens. The spontaneous ignition activation energy was calculated from the dependence of the ignition time on the inverse value of thermodynamic temperature (Semenov method). This dependence was measured in the hot-air (Setchkin) furnace according to ISO 871: 2006.

MARTINKA *et al.* (2016) continued the study of Norway spruce wood with investigation of the way heat treatment of Norway spruce wood influence on the heat release rate and propensity for fire propagation in the flashover phase. The heat release rate was determined by a cone calorimeter in accordance with ISO 5660-1:2002. The propensity for fire propagation in the flashover phase was determined from the cone calorimeter. Three kinds of samples were used: heat untreated, heat treated in accordance with the ThermoWood – Thermo-S programme (maximum temperature during the heat treatment was 190 °C) and heat treated in accordance with the ThermoWood – Thermo-D programme (maximum temperature during the heat treatment was 212 °C). Obtained results proved that heat treatment of spruce wood has no impact on the propensity for fire propagation in the flashover phase. The time interval to reach the flashover phase of both heat treated and untreated spruce wood specimens was from 120 to 600 seconds. The heat treatment of spruce wood by both used thermal programmes caused a significant decrease in the heat release rate.

With the evaluation of combustion kinetics of Norway spruce stem wood effects in relation to the dry and wet torrefaction dealt BACH *et al.* (2017). They thermogravimetrically studied the thermal reactivity of the woods in nitrogen and air to simulate pyrolysis and combustion conditions, followed by a kinetic evaluation employing multi-pseudo-component models. The results show that dry torrefaction has unpronounced effects on the activation energy of cellulose and lignin in the subsequent thermal conversion processes, while these figures increase after wet torrefaction. In addition, wet and dry torrefaction show opposite trends in the char combustion: while dry torrefaction increases both the activation energy and pre-exponential factor of char, wet torrefaction decreases these kinetic parameters.

For the calculation of the activation energy of material decomposition is possible to use several methods. MARTINKA *et al.* (2015b) tested three iso-conversion three methods that are used most often in practice. Specifically, it was the Kissinger method, Flyn-Wall-Ozawa method and Kissinger-Akahira-Sunos method. As most of the methods by which it is possible to calculate the activation energy of the decomposition, those are also based on the Arrhenius equation.

This paper deals with the study of the activation energy needed to spontaneously ignite / flash ignite the Norway spruce wood and Norway spruce Thermowood chemically treated, applying the analogy of the Arrhenius equation, published by SEMENOV in 1959, which is based on using the results of thermal analysis of the materials considered.

EXPERIMENT

The specimens tested were produced from the Norway spruce wood and Thermowood.

The Norway spruce wood was obtained during the winter timber harvesting in the territory of the University Forest Enterprise in 2016 and was processed in the Development Workshops and Laboratories of the Technical University in Zvolen in the spring of the same year. Thermowood, according to REINPRECHT and VIDHOLDOVÁ (2011), is produced by the the thermal modification of the wood at a temperature of 150–260 °C. In this process, the chemical structure of the wood is changed, too.

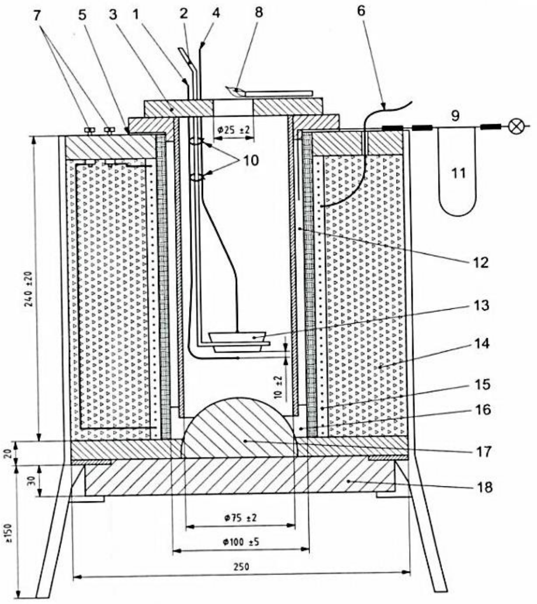
The Norway spruce Thermowood for testing was obtained from the company SLOV-DV Ltd., settled in Galanta, Slovakia. It was chemically treated using the vacuum-pressure impregnation of wood. Wood was modified to be resistant against the decaying fungi, pests and the influence of changing atmospheric conditions.

The first step of the procedure was to determine the ignitability of specimens tested. For that reason, the incendiary hot-air oven was used and the methodology for testing the spontaneous ignition and flash point temperature according to the STN ISO 871 standard was applied.

The incendiary hot-air oven consists of the electric heating unit and specimen holder (Figure 1).

As a measuring device with thermocouples for measurement of temperature course withing the specimens and on their surface, the ALMEMO 2290-8 device was used.

The tests were carried out on specimens of Norway spruce wood and Thermowood. Each specimen weighted 3.0 ± 0.2 g and conditioned at temperature of 23 ± 2 °C and a relative humidity of 50 ± 5 %, for 40 hours, according to the STN EN ISO 291 standard.



1	Thermocouple TC_2
2	Support rod of specimen holder
3	Fire-proof circular cover
4	Thermocouple TC_1
5	Sealing ring
6	Thermocouple TC_3
7	Heating coil terminals
8	Ignition flame
9	Air supply
10	Metal fixing clips
11	Flowmeter
12	Tangential air flow to the cylinder
13	Specimen pan
14	Mineral fibre wool
15	Threaded spiral made of chromium-nickel
in	heat durable cement
16	Three spacer rings for location of the inner cylinder and its support
17	Removable control cup
18	Removable thermal insulation

Fig. 1 Setchkin Furnace (ASTM INTERNATIONAL).

The specimens with moisture of 9 % were modified before the weighing, cut to prisms with dimension 20 × 20 × 10 mm. The lower weight variation as compared to the STN ISO 871 standard was chosen to limit the uncertainty of measurement resulting from the dependency between the induction period and the weight.

Flash point temperature determination

The airflow rate in the incendiary hot-air oven was set to 25 $\text{mm}\cdot\text{s}^{-1}$, adjusting the actual airflow rate qv over the entire cross section of inner cylinder to the computed value (in liters per minute) using the following equation (STN ISO 871:2010):

$$q_v = 6.62 \times T \tag{1}$$

where: T – Thermodynamic temperature (K), qv – Airflow rate ($\text{mm}\cdot\text{s}^{-1}$).

Airflow rate in the incendiary hot-air oven was maintained within ± 10 % of the computed value. The specimen was placed in the incendiary hot-air oven. After starting the timing device, there was ignited a flame and the appearance of moderate flash ignition or explosion of combustible gases was observed, optionally followed by a continuous combustion of the specimen, and also flaming or glowing, when the temperature is suddenly increasing. After 10 minutes the temperature is reduced or raised to 50 °C, depending on whether the flash ignition has occurred or not. If not, the test is repeated with a new specimen. When the range for flash point was set, the tests started at a temperature of 10 °C lower than the highest temperature within the temperatures range, and continued with reducing the temperature by 10 °C to the temperature at which flash ignition did not occurred during 10 minutes period.

The lowest air temperature at which, during the 10 minutes, the flame was observed was recorded as a flash point temperature.

Spontaneous ignition temperature determination

The procedure was the same as by the flash point temperature determination, but without application of the igniting flame. Spontaneous ignition is evident during the combustion of specimen by flame or glowing. It is difficult visually to determine it, because the combustion is manifesting rather by glowing than by flame. Therefore, faster temperature rise in the thermocouple accompanied by visual observation is considered for more reliable evidence.

The lowest air temperature at which the specimen was ignited within 10 minutes was recorded as the spontaneous ignition temperature.

Spontaneous ignition and flash point activation energy determination

Determination of the necessary spontaneous ignition temperatures with functional dependencies of induction periods was carried out applying the Setchkin Furnace test, i.e. the thermoanalytical methods.

First, the minimum temperatures of initiation were derived. After their finding, the induction periods were tested of each about 10 °C higher than in the previous measurement, in the end to get a total of 10 measurements required for the dependency analysis.

The dependency analyses between the induction periods and the inverse values of thermodynamic temperature for both specimen types were performed in Statistica 8 software. In the same software was derived the exponencial equation, from which the pre-exponential factor was used to calculate the activation energy of the process of spontaneous ignition / flash point.

Before the experiment itself, the temperature in the incendiary hot-air oven was adjusted to match the estimated value of the spontaneous ignition or flash point of the Norway spruce wood and Thermowood. According to the temperature set, the airflow was adjusted to the air flow at a constant rate. Using a mirror, positioned above the oven, and stopwatch, there had been observing the moment and time of the spontaneous ignition / flash point, which should start, according to the standard, in 10 minutes (600 seconds).

If the spontaneous ignition / flash point had not occurred, the testing was repeated with a new specimen at a gradually increasing temperature by 10 °C until the specimen was not spontaneously / flash ignited in 10 minutes. This procedure was repeated until the minimum temperature of spontaneous ignition / flash point was found, which was also recorded with accurate time information. After determining the minimum spontaneous / flash point temperature, the temperature was gradually increased every 10 °C until reaching about 100 °C higher temperature as the minimum spontaneous / flash ignition temperature determined.

Activation energy calculation

The calculation of activation energy of spontaneous ignition / flash ignition was performed according to equation (2), published by SEMENOV (1959), that is an analogy to the equation Arrhenius.

$$E = ln\left(\frac{\tau}{A}\right) \times R \times T \tag{2}$$

where:

τ – Induction period of spontaneous ignition / flash ignition (s)
A – Pre-exponential (frequency) factor (-)
E – Activation energy of spontaneous ignition / flash ignition ($J \cdot mol^{-1}$)
R – Gas constant (8.314 $J \cdot K^{-1} \cdot mol^{-1}$)
T – Ignition termodynamic temperature (K)

RESULTS AND DISCUSSION

The measured data together with the temperature recalculated values (inverse value of the temperature in °C to the thermodynamic temperature in K, necessary for the calculation of the activation energy) are shown in the tables 1–4. It is evident, that with increasing values of particular temperatures, the values of induction periods were decreasing. With increasing thermal loading, the specimen resisted to thermal action a shorter time.

Spontaneous ignition temperatures and induction period determination

In case of Norway spruce wood specimens, the minimum spontaneous ignition temperature value of 429 °C (702 K) was recorded at the first measurement. It was reached in 396 s from the start of the test. During the test, the values of induction period were decreasing with increasing temperature values. The minimum time (182 s), the Norway spruce wood resisted to temperature of 472 °C (745 K) at the last measurement (Table 1).

Tab. 1 Spontaneous ignition temperatures of Norway spruce wood.

Measurement no.	Induction period τ (s)	Temperatures average t (°C)	Temperatures average T (K)	Temperature inverted value 1/T (K^{-1})
1.	396	429	702	0.001424
2.	353	437	710	0.001407
3.	371	450	723	0.001382
4.	297	442	715	0.001398
5.	249	445	718	0.001391
6.	247	451	724	0.001381
7.	207	455	728	0.001373
8.	212	462	735	0.001360
9.	198	467	740	0.001349
10.	182	471	744	0.001343

Table 2 shows an overview of the temperatures reached, completed with the induction periods. The lowest spontaneous ignition temperature of 418 ° C (691 K) reached the Thermowood specimen right at the first measurement. In average time of 360 s, there occurred its spontaneous ignition in the absence of ignition open sources. With increasing temperature, the specimen resisted to high temperatures for a shorter time.

Tab. 2 Spontaneous ignition temperatures of Thermowood.

Measurement no.	Induction period τ (s)	Temperatures average t (°C)	Temperatures average T (K)	Temperature inverted value 1/T (K^{-1})
1.	360	418	691	0.001447
2.	332	427	700	0.001427
3.	285	428	701	0.001426
4.	239	431	704	0.001419
5.	223	443	716	0.001395
6.	221	448	721	0.001387
7.	188	457	730	0.001369
8.	185	461	734	0.001361
9.	187	457	730	0.001368
10.	176	446	719	0.001389

Flash point temperatures and induction period determination

The lowest flash point temperatures, at which the Norway spruce wood developed as much combustible gases, that those in the mixture with air, under the action of open flame were flash ignited, was recorded with a value of 355 °C (628 K) at the first measurement, and at a the time of 587 s.

The minimum flash point temperature in case of the Thermowood specimen was found in time of 538 s, i.e. 346 °C (619 K). The shortest time (256 s) resisted the specimen of Thermowood to the temperature of 415 °C (688 K), which was also the maximum flash point temperature, measured at the last measurement.

Tab. 3 Flash point temperatures of Norway spruce wood.

Measurement no.	Induction period τ (s)	Temperatures average t (°C)	Temperatures average T (K)	Temperature inverted value 1/T (K^{-1})
1.	587	354	627	0.001593
2.	505	363	636	0.001571
3.	470	371	644	0.001553
4.	443	379	652	0.001532
5.	390	385	658	0.001519
6.	399	393	666	0.001499
7.	359	395	668	0.001496
8.	351	404	677	0.001476
9.	309	410	683	0.001463
10.	278	418	691	0.001446

Tab. 4 Flash point temperatures of Thermowood.

Measurement no.	Induction period τ (s)	Temperatures average t (°C)	Temperatures average T (K)	Temperature inverted value 1/T (K^{-1})
1.	538	345	618	0.001616
2.	490	355	628	0.001592
3.	457	362	635	0.001575
4.	365	369	642	0.001558
5.	374	378	651	0.001536
6.	348	385	658	0.001518
7.	308	393	666	0.001499
8.	286	400	673	0.001484
9.	284	407	680	0.001471
10.	256	415	688	0.001453

The spruce wood specimens achieved correlation value of 0.85, which represents a relatively high dependence.

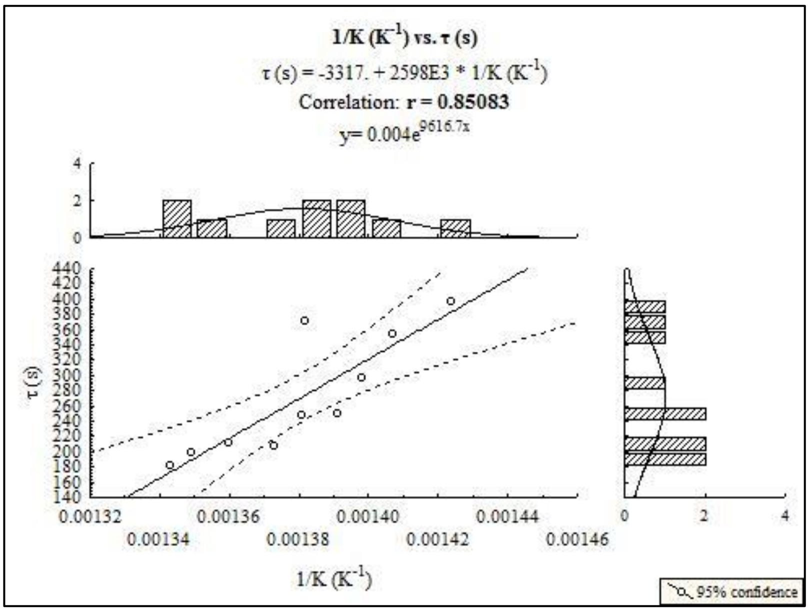


Fig. 2 Graph of dependency between the spontaneous ignition induction period and the inverse value of thermodynamic temperature of Norway spruce wood.

In Figure 3 are introduced the results of the dependency analysis for the Thermowood

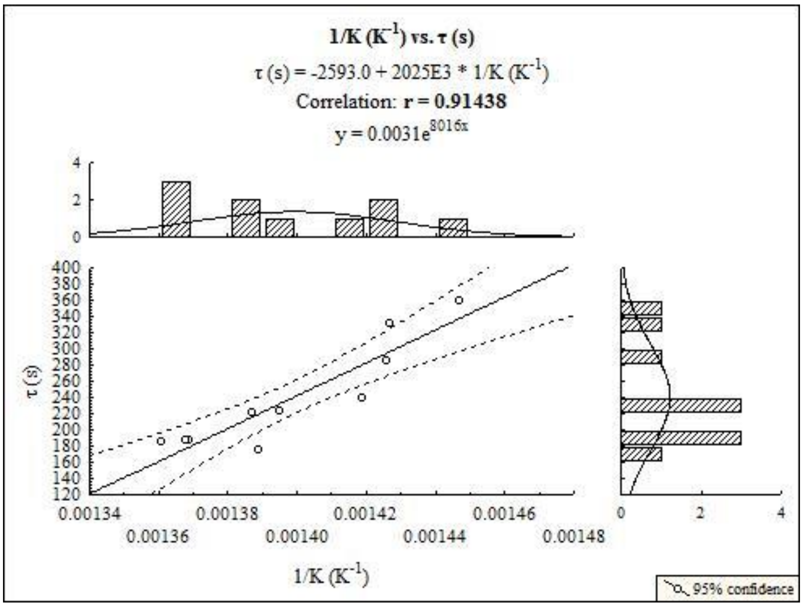


Fig. 3 Graph of dependency between the spontaneous ignition induction period and the inverse value of thermodynamic temperature of Thermowood.

Specimens prepared from Norway spruce wood and Thermowood showed in the dependency between the flash point induction period and the inverse value of the thermodynamic temperature very high value of the correlation coefficients: r = 0.98 for Norway spruce wood and r = 0.97 for Thermowood.
Figure 4 shows result of the dependency analysis between the flash point induction period and the inverse value of the thermodynamic temperature, which is the most evident in thermodynamic temperature inverse value of 0.001593 at temperature of 345 °C, which the Norway spruce wood specimen reached in 587 s from the beginning of the measurement.

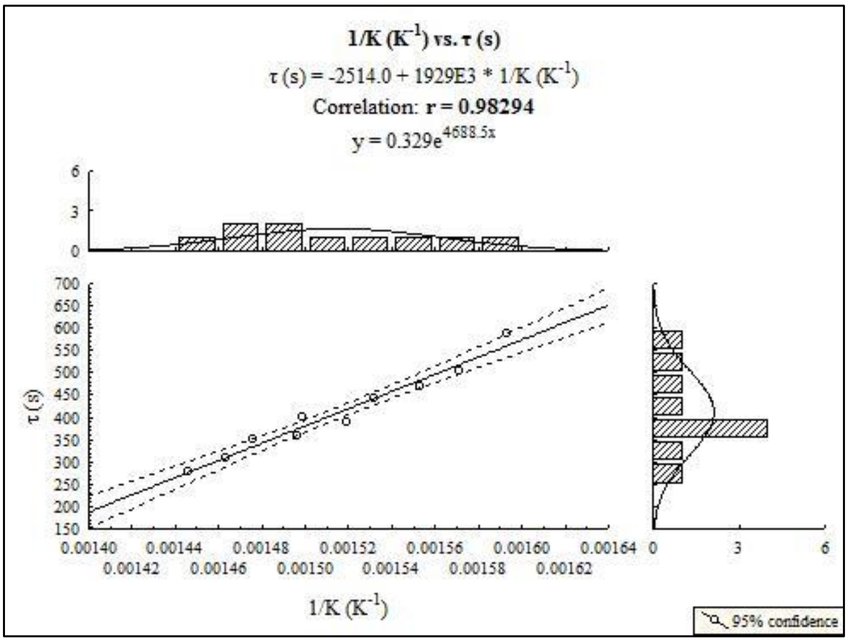


Fig. 4 Graph of dependency between the flash point induction period and the inverse value of thermodynamic temperature of Norway spruce wood.

Figure 5 introduces result of the dependency analysis between the flash point induction period and the inverse value of thermodynamic temperature of Thermowood.

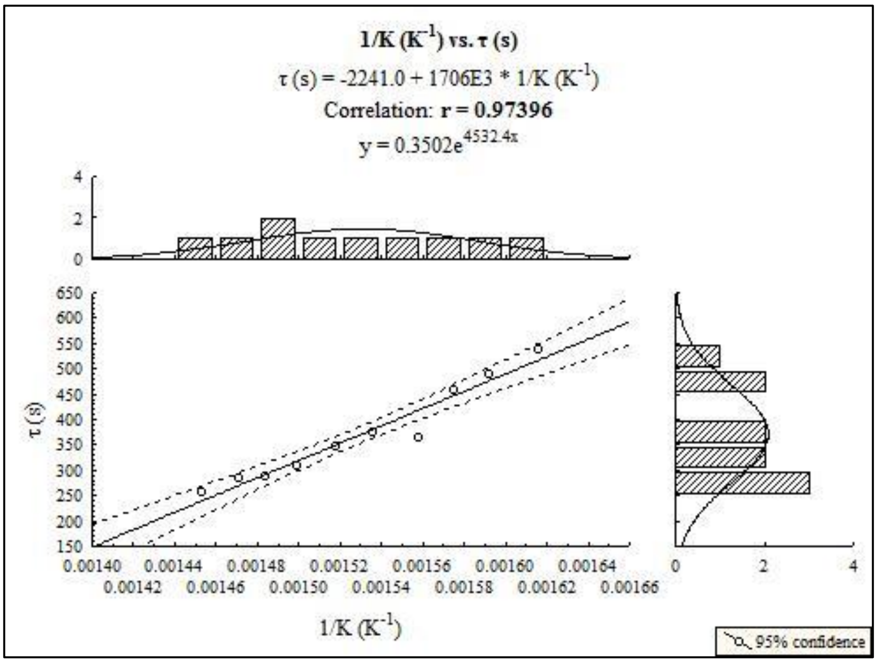


Fig. 5 Graph of dependency between the flash point induction period and the inverse value of thermodynamic temperature of Thermowood.

Substituting the measured values from the relevant tables and derived pre-exponential factors from exponential equations into the equation for calculation the activation energy, there were calculated the particular values of activation energy of specimens tested (table 4).

Tab. 4 Result values of activation energy for spontaneous ignition / flash point of tested wood specimens.

Specimen	Activation energy of spontaneous ignition temperature ($J \cdot mol^{-1}$)	Activation energy of flash point temperature ($J \cdot mol^{-1}$)
Norway spruce wood	67.15	39.09
Thermowood	67.01	37.75

The experiments results confirmed the fact that the activation energy is independent of the temperature. It is generally known that the activation energy decreases with increasing temperature, in the simplest situations the dependence on temperature is linear.

The values of the minimum activation energy of spontaneous ignition as well as flash point of specimens are higher in case of Norway spruce wood. Therefore, it was also required higer energy value of thermal action, in order to start the process of spontaneous ignition or flash ignition.

Untreated Norway spruce wood showed the highest values of activation energy of spontaneous ignition and flash point of all specimens tested. In this specific case, it is also possible to evaluate the specimens made of Norway spruce wood to have the highest resistance to thermal loading, using the available testing apparatus.

We compared the results we achieved with the results of other authors dealing with this issue. First we compared our results with results achieved by MATEJOVA (2012). She presented the activation energy of spontaneous ignition of 62 $kJ \cdot mol^{-1}$ (the difference in comparison to our measurement is of 5.2 kJ·mol^{-1}) and of flash ignition of 28.5 kJ·mol^{-1} (the difference is of more than 10 kJ·mol^{-1}) for the set of specimens of Norway spruce wood.

In accordance to the study of MARTINKA *et al.* (2015a) the lowest spontaneous ignition activation energy 44.1 kJ·mol^{-1} was measured for the dust form and the highest activation energy 59 kJ·mol^{-1} was measured for the pellet form of spruce wood. Our experiment results with wood showed the activation energy of about 67 kJ·mol^{-1}, what can be considered for correct value, when compared to the results of MARTINKA (2015a) and also MATEJOVA (2012). Thus, the obtained results proved that the form of spruce wood has a significant influence on its spontaneous ignition activation energy. Further obtained results proved that the activation energy of spontaneous ignition is almost independent on the temperature.

For comparison, there are introduced also the results achieved by MATSUO *et al.* (2016), who studied primary the color changes of four softwood and seven hardwood species during hygrothermal treatment. Those were compared among species, but also kinetically evaluated. In the kinetic analysis they used the time-temperature superposition principle, which uses the whole data set. The apparent activation energies were set to 24.3–40.8 kJ·mol^{-1} for softwood and 32.3–61.3 kJ·mol^{-1} for hardwood. The average apparent activation energy for hardwood was higher than for softwood. These values were lower than those calculated from other material properties.

The values of spontaneous ignition / flash point temperature were further compared with the results of MITTEROVÁ *et al.* (2012). In this comparison was noticed a difference in the value of determined spontaneous ignition temperature of Norway spruce wood. MITTEROVÁ *et al.* (2012) introduced the value of 460 °C while the value measured in our experiment was significantly lower (429 °C).

As an additional reference data source, there were used the results obtained by JANEČEK (2013). In this case was found a difference in the flash point temperature of Norway spruce wood. The reference value of the flash point temperature was of 378 °C. The flash point temperature measured in our experiment was of 355 °C, i.e. was lower of 23 °C.

The reason of the differences in the results achieved in the studies mentioned above could be the different age of the wood, its moisture or the other parameters which were not included in the comparison of the results achieved, because the information is not available or was not considered to be essential for testing.

CONCLUSIONS

To understand the combustion process, it is necessary to distinguish among the particular fire fire or thermal parameters of materials and to understand their significance in the combustion process. Among those parameters belong also the activation energy, which has a significant influence on combustion process, fire initiation and which influences the thermal degradation process of specific materials.

The results obtained within particular experiments showed that the spontaneous ignition temperature of Norway spruce wood specimens varied in the temperature range of 429 °C to 472 °C. The results achieved by the Thermowood specimens were comparable to Norway spruce wood specimens results (temperature range of 418 °C to 462 °C). The minimum flash point temperature was reached at temperature of 346 °C (619 K) in case of the Thermowood specimens. The Norway spruce wood specimen flash point has occurred at the temperature about 9 °C higher, i.e. 355 °C (628 K). The activation energy needed to reach the spontaneous ignition was very similar for both Norway spruce specimen kinds tested, i.e. 67.15 kJ·mol^{-1} in case of untreated Norway spruce wood and 67.01 kJ·mol^{-1} in case of Thermowood. The more significant difference showed the results of activation energy calculation based on the data on flash ignition, where the untreated Norway spruce wood reached the value of 39.09 kJ·mol^{-1} and the Termowood of 37.75 kJ·mol^{-1}.

The dependency analyses between the induction periods and the inverse values of thermodynamic temperature for both specimen types showed very strong dependency between the variables tested in all tests.

These results achieved in all of the experiments realised will be further used as input parameters in the modelling the behaviour of compartment fires. They are immediately applicable also for planing the necessary fire safety measures and design of modern structures, especially the wooden ones.

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ACKNOWLEDGEMENT

This work was supported by the Slovak Research and Development Agency under the conctract No. APVV-0057-12 (50 %) and No. APVV-16-0326 (50 %).

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