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THE ANALYSIS OF MASS LOSS AND ACTIVATION ENERGY OF SELECTED FAST-GROWING TREE SPECIES AND ENERGY CROPS USING THE ARRHENIUS EQUATION

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ABSTRACT

Biomass as a renewable energy source covers the energy needs of the state to a great extent. In addition to the knowledge of the available sources of biomass for the energy use, the key information on thermal properties and fire performance would contribute to further understanding of their energy properties as well as the combustion process. The aim of this analysis was to calculate the activation energies of selected fast-growing tree species (*Paulownia tomentosa*, *Salix viminalis* variety Tora, *Populus x euroamericana* variety MAX 4) and energy crops (*Sida hermaphrodita*, *Arundo donax*, *Miscanthus x giganthus*)., Spontaneous ignition temperature and induction time must be estimated to calculate the activation energy. Spontaneous ignition temperatures and induction time were determined by the Setchkin Furnace test in accordance with the standard STN ISO 871. The analyses were completed with the mass loss analysis. The mass loss of individual samples was determined by using a non-standard testing method. The values of the activation energy gathered in the analysis showed that activation energy values in all tested samples were similar, in the interval of 136.72 ± 7.28 $kJ \cdot mol^{-1}$. The differences in the development of mass loss of fast-growing tree species and energy crop samples were found. The mass loss course of the fast-growing tree species was more linear, while the mass loss development of energy crops could be divided into two stages.

Key words: activation energy, spontaneous ignition, mass loss, energy crop, fast-growing tree species.

INTRODUCTION

In order to understand the combustion process, it is necessary to distinguish between the fire performance and thermal parameters of materials. Moreover, their significance in the combustion process must be understood. The activation energy having a significant influence on combustion process, fire initiation and affecting thermal degradation process of specific materials is one of those parameters, too. There are several methods to calculate the activation energy. Simple methods (e.g. Arrhenius equation) to much more complex methods (e.g. Ozawa-Flynn-Wall, Kissinger-Akahira-Sunos or Friedman, ASTM-E698-05) requiring the results of thermogravimetry, differential thermogravimetry and differential scanning calorimetry and other progressive instruments and methodologies result in calculating the activation energy and the combustion process kinetics in a complex and precise way. The progressive approaches to the analysis of biomass combustion kinetics are evident also in the studies of several foreign and domestic authors.

The thermal behaviour of energy crops *Arundo donax* and *Miscanthus x giganthus* was studied by JEGUIRIM, DORGE and TROUVÉ (2010). Thermogravimetric analyses were carried out at 5 °C·min^{-1} under air atmosphere. The parameters of reacton kinetics, i.e. activation energy were calculated using the Arrhenius equation.

KOK AND ÖZGÜR (2013) focused the research on combustion behaviour of agricultural residues nown as miscanthus, poplar wood and rice husk. They investigated them using the thermal analysis technique. The differential scanning calorimetry (DSC) and thermogravimetry (TG-DTG) techniques were used. Combustion experiments were performed in five different heating rates (5, 10, 15, 25 and 50 °C·min^{-1}). The reaction regions, ignition and burnout temperatures, heat flow rate values of biomass samples were determined. Activation energy of the biomass samples was calculated using three different iso-conversional methods. It was observed that all three agricultural residues show similar combustion characteristics.

The wild cane thermochemical properties were studied by APONTE *et al*. (2013). Those properties were obtained using ultimate, proximate, and thermogravimetric analyses (TGA). The thermogravimetric analyses were carried out using N_2 as carried gas and under different heating rates (β = 10, 20 and 35 °C·min^{-1}). The activation energy was estimated based on TGA data and using the isoconversional method.

The pyrolysis and combustion energetic characterisation of coal, biomass and mixture samples were studied by AGARWAL ET AL. (2014). The thermogravimetric analyser (STA) and microscale combustion calorimeter (MCC) are used for this purpose. The STA was used to measure the gravimetric and energetic response of pyrolyzing sample under inert atmosphere. The MCC was used to quantify the dynamic heat output from combustion of the gases produced during the pyrolysis process. They found that the co-pyrolysis of coal and biomass fuel mixtures exhibited a weighted additive gravimetric and energetic behaviour in terms of pyrolysis and combustion aspects, respectively.

CHEN *et al*. (2015) investigated the pyrolysis characteristics and kinetic of five lignocellulosic biomasses. The biomass samples were represented by pine wood sawdust, fern (*Dicranopteris linearis*) stem, wheat stalk, sugarcane bagasse and jute (*Corchorus capsularis*) sticks. They were investigated using thermogravimetric analysis. The pyrolysis of five lignocellulosic biomass was divided into three stages corresponding to the pyrolysis of hemicellulose, cellulose and lignin, respectively. The single Gaussian activation energy distributions were determined. The kinetic parameters of different stages were used as initial guess values for three-parallel-DAEM model calculation with good fitting quality and fast convergence rate. The mass fractions of hemicellulose, cellulose and lignin in lignocellulosic biomass were estimated.

AHMAD *et al*. (2017) provided the thermogravimetric analyses revealed the bioenergy potential of *Eulaliopsis binate*. The plant biomass was subjected to thermal degradation experiments at three heating rates: 10, 50 and 50 K·min^{-1}. The kinetics analyses were performed through isoconversional models of Kissinger-Akahira-Sunos and Ozawa-Flynn-Wall, followed by the calculation of thermodynamic parameters of activation.

CAI *et al*. (2018) focused on processing the thermogravimetric analysis data for isoconventional kinetics analysis of lignocellulosis corn stalk biomass pyrolysis. They reviewed the overall procedure of processing TGA data for isoconversional kinetic analysis of lignocellulosic biomass pyrolysis by using the Friedman isoconversional method. This includes the removal of "error" data points and dehydration stage from original TGA data, transformation of TGA data to conversion data, differentiation of conversion data and smoothing of derivative conversion data, interpolation of conversion and derivative conversion data, isoconversional calculations, and reconstruction of kinetic process. The detailed isoconversional kinetic analysis of TGA data gathered in the pyrolysis of corn stalk at five heating rates were presented.

In Slovak conditions, the thermal properties of woody biomass were studied by several authors. They used especially the Arrhenius equation to calculate the activation energy of woody biomass.

MARTINKA *et al.* (2015) focused on the influence of spruce wood form on ignition activation energy.

MARTINKA *et al.* (2017) carried out the research into initiatory parameters of poplar wood (Populus tremula L.). The initiatory parameters (critical heat flux density and surface temperature in the time of initiation) were set on a conical calorimeter using a testing procedure in accordance with ISO 5660-1:2015.

MARTINKA *et al.* (2018) investigated the impact of heat flux on fire risk of the selected fast-growing woods, i.e. hybrid poplar J-105 (Populus nigra x P. maximowiczii), white willow (Salix alba L.) and black locust (Robinia pseudoacacia L.) woods. The heat of combustion was determined by a bomb calorimeter. Fire risk was evaluated with a cone calorimeter at different heat fluxes.

DZURENDA, PNAKOVIČ (2016) studied the influence of the burning temperature evaluated for the nonvolatile combustible content of wood and bark of plantation-grown trees, at the temperature ranging from 500 °C to 1000 °C, in relation to ash production and concentration of Ca, Mg, K, Mn, Zn, and Fe in ash, thermal properties and ash fusibility.

RANTUCH *et al.* (2016) used the Arrhenius equation to calculate the ignition activation energy of materials based on polyamide 6.

KAČÍK, ĎURKOVIČ, KAČÍKOVÁ (2012) studied and published chemical profiles of wood components of poplar varieties for their energy utilization.

KAČÍK *et al.* (2017) estimated and published the results of the thermal analysis of heat-treated silver fir wood and larval frass.

ZACHAR *et al*. (2017) published the results of the analysis focusing on the activation energy required for spontaneous ignition and flash point of the Norway spruce and thermowood specimens.

LUPTÁKOVÁ *et al*. (2018) presented comparison of activation energy of thermal degradation of heat sterilised silver fir wood to larval frass regarding fire safety. The ignition activation energies of wood samples using the Arrhenius equation were calculated as well.

The aim of this analysis is to calculate the activation energies of selected fast-growing tree species (*Paulownia tomentosa, Salix viminalis* variety Tora, *Populus x euroamericana* variety MAX 4) and energy crops (*Sida hermaphrodita*, *Arundo donax*, *Miscanthus x giganthus*). Firstly, the spontaneous ignition temperatures were estimated in order to calculate the energies.

Those data were completed with data of sample mass loss estimated using the non-standard method of testing.

EXPERIMENTAL PART

The test samples of fast-growing tree species *Salix viminalis* variety Tora and *Populus x euroamericana* variety MAX 4 were taken from the existing plantations of the University Foret Enterprise of the Technical University in Zvolen territory.

The test samples of fast-growing tree species *Paulownia tomentosa*, and energy crops *Sida hermaphrodita*, *Arundo donax, Miscanthus x giganthus* were taken from the plantations belonging to the Agricultural Co-operative Dolné Saliby.

Each energy crop sample consisted of a mixture of the crop small particles and weighed 3.0 ± 0.05 g, according to the testing method requirements. The samples of fast-growing tree species were cut to blocks with dimensions of 20 × 20 × 10 mm to be used for spontaneous ignition temperature examination and blocks with dimensions of 50 × 40 × 20 mm for mass loss analysis. The moisture content of all tested wood and crop samples was 12 %.

The ash content of the fast-growing tree species and energy crops, the elemental analysis and gross calorific values of the samples tested has already been published in *Zachar et al.* (2018).

Before the tests, all the samples were dried in hot-air oven to the moisture content of 12 % and further conditioned at the temperature of 23 ± 2 °C and relative humidity of 50 ± 5 %, for 40 hours according to requirements of the sstandard STN EN ISO 291. Totally, three samples of each fast-growing tree species and energy crop were tested.

It was necessary to estimate the temperature of spontaneous ignition to calculate the activation energies. Incendiary hot-air oven was used and the methodology for testing the spontaneous ignition temperature according to STN ISO 871 was applied in order to determine it.

The lowest air temperature at which the sample was ignited within 10 minutes was recorded as the spontaneous ignition temperature. Subsequently, the induction time was found.

The dependency analyses between the induction time and the inverse values of thermodynamic temperature for the samples were performed in the software Statistica 12. The exponencial equation was derived in the same software. The pre-exponential factor was further used to calculate the activation energy of spontaneous ignition.

The calculation of activation energy of spontaneous ignition (kJ·mol^{-1}) was performed according to the equation (2), that is an analogy to the Arrhenius equation.

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

where: τ – Inductiontime of spontaneous ignition (s)

A – Pre-exponential (frequency) factor (-)

E – Activation energy of spontaneous ignition (J·mol^{-1})

R – Gas constant (8.314 J·K^{-1}·mol^{-1})

T – Ignition termodynamic temperature (K)

In calculation of the activation energy only the mean values of the spontaneous ignition temperatures and induction time were involved.

The non-standard method of solids thermal properties testing was applied to analyse the mass loss of the samples. The samples of fast-growing tree species and energy crops were exposed to thermal loading by a radiant heater with the power of 1,000 W, situated in the distance of 3 cm far from the sample, for a specific time, i.e. fast-growing tree species samples for 600 s and energy crops samples for 300 s. The mass loss of the samples (g) was measured and recorded each 10 s.

RESULTS AND DISCUSSION

The results gathered in the performed analysis are introduced in this part of the paper. Firstly, the results associoated with the spontaneous ignition temperatures and induction time, the results of activation energy calculation and finally, the mass loss analysis results are presented.

Spontaneous ignition temperature analysis results

The spontaneous ignition temperatures, 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 Tab. 1–6.

Tab. 1 shows an overview of the temperatures reached by *Paulownia tomentosa* completed with the induction time data.

Tab. 1 Spontaneous ignition temperatures of *Paulownia tomentosa*

Measurement no.	Induction time τ (s)	Spontaneous ignition temperature t (°C)	Spontaneous ignition temperature T (K)	Temperature inverted value 1/T (K^{-1})
1.	332.00	420.10	693.25	0.001442
2.	292.00	410.98	684.13	0.001462
3.	263.20	441.87	715.02	0.001399
Mean	295.70	424.32	697.47	0.001434

Tab. 2 shows an overview of the temperatures reached by *Salix viminalis* variety Tora completed with the induction time data.

Tab. 2 Spontaneous ignition temperatures of *Salix viminalis* variety Tora

Measurement no.	Induction time τ (s)	Spontaneous ignition temperature t (°C)	Spontaneous ignition temperature T (K)	Temperature inverted value 1/T (K^{-1})
1.	338.20	412.65	685.80	0.001458
2.	303.40	426.63	699.78	0.001429
3.	345.00	419.09	692.24	0.001445
Mean	328.87	419.46	692.61	0.001444

Tab. 3 shows an overview of the spontaneous ignition temperatures and induction time reached by *Populus x euroamericana* variety MAX 4.

Tab. 3 Spontaneous ignition temperatures of *Populus x euroamericana* variety MAX 4

Measurement no.	Induction time τ (s)	Spontaneous ignition temperature t (°C)	Spontaneous ignition temperature T (K)	Temperature inverted value 1/T (K^{-1})
1.	339.00	424.92	698.07	0.001433
2.	341.40	417.14	690.29	0.001449
3.	309.40	419.69	692.84	0.001443
Mean	329.90	420.58	693.73	0.001442

Tab. 4 shows an overview of the spontaneous ignition temperatures and induction time reached by *Sida hermaphrodita.*

Tab. 5 shows an overview of the spontaneous ignition temperatures and induction time reached by *Arundo donax.*

Tab. 4 Spontaneous ignition temperatures of *Sida hermaphrodita*

Measurement no.	Induction time τ (s)	Spontaneous ignition temperature t (°C)	Spontaneous ignition temperature T (K)	Temperature inverted value 1/T (K^{-1})
1.	212.80	434.67	707.82	0.001413
2.	222.10	430.56	703.71	0.001421
3.	209.30	426.44	699.59	0.001429
Mean	214.70	430.56	703.71	0.001421

Tab. 5 Spontaneous ignition temperatures of *Arundo donax*

Measurement no.	Induction time τ (s)	Spontaneous ignition temperature t (°C)	Spontaneous ignition temperature T (K)	Temperature inverted value 1/T (K^{-1})
1.	215.00	431.67	704.82	0.001419
2.	206.40	428.70	701.85	0.001425
3.	212.80	433.24	706.39	0.001416
Mean	211.40	431.20	704.35	0.001420

Tab. 6 shows an overview of the spontaneous ignition temperatures and induction time reached by *Miscanthus x giganthus.*

Tab. 6 Spontaneous ignition temperatures of *Miscanthus x giganthus*

Measurement no.	Induction time τ (s)	Spontaneous ignition temperature t (°C)	Spontaneous ignition temperature T (K)	Temperature inverted value 1/T (K^{-1})
1.	234.60	426.78	699.93	0.001429
2.	228.70	433.71	706.86	0.001415
3.	231.30	416.22	689.37	0.001451
Mean	231.50	425.57	698.72	0.001431

The mean spontaneous ignition temperature values of all samples were in the range of 425.28 ± 4.90 °C (698.43 ± 4.90 K). The lowest mean spontaneous ignition temperature value was recorded by *Salix viminalis* variety Tora (419.46 °C, i. e. 692.61 K) reached in 328.87 s from the start of the test. The highest mean spontaneous ignition temperature value was found at *Arundo donax* (431.20 °C, i.e. 704.35 K), which showed the shortest mean induction time value, too. The longest mean induction time was measured by *Populus x euroamericana* variety MAX 4 (329.90 s).

The results also showed that the energy crops reached higher spontaneous ignition temperatures (429.11 ± 3.08 °C, i.e. 702.26 ± 3.08 K) and shorter induction time (219.13 s on average) than the fast-growing tree species (mean spontaneous ignition temperature of 421.45 ± 2.55 °C, i.e. 694.6 ± 2.55 K) and induction time of 318.16 s on average.

Activation energy analysis results

The results of activation energy calculation using the Arrhenius equation are introduced.

Tab. 7 shows the values of pre-exponential factor (*A*) representing the regression coefficient in the correlation equation calculated between the spontaneous ignition temperature and induction time values of individual samples. Those data were used in calculation of the activation energy of individual samples, including the mean values of spontaneous ignition temperature and induction time. The calculated activation energy values are shown in Tab. 7.

Tab. 7 Activation energy values of individual tested samples.

Sample	Pre-exponential factor (*A*)	Activation energy of spontaneous ignition ($kJ \cdot mol^{-1}$)
Paulownia tomentosa	0.00647	127.90
Salix viminalis variety Tora	0.00368	136.90
Populus x euroamericana variety MAX 4	0.00175	145.60
Sida hermaphrodita	0.00215	136.00
Arundo donax	0.00104	144.30
Miscanthus x giganthus	0.00433	129.60
Mean	-	136.72

The activation energies of fast-growing tree species and energy crops were similar. The mean for all of them was of 136.72 ± 7.28 $kJ \cdot mol^{-1}$. Those results confirm the suitability of energy crops to be used as a renewable energy source. Their advantage in comparison to fast-growing tree species is their higher and mostly annual yields with very similar energy properties.

Mass loss analysis results

For better understanding the thermal decomposition process, it is recommended to perform mass loss analysis. The TG, DTG analyses are performed to achive more precise results. However, simplified approach based on non-standardized method allowing studying the material sample mass loss in time was used in this analysis. The TG and DTG analyses allow studying mass loss during the sample heating prior to specified temperature interval.

The individual samples mass loss developments are introduced in Fig. 1–6. Those are also completed with data on the samples time of autoignition and flame extinguishing, when exposed to the thermal loading for 5 (crops) – 10 min (trees), (Fig. 7).

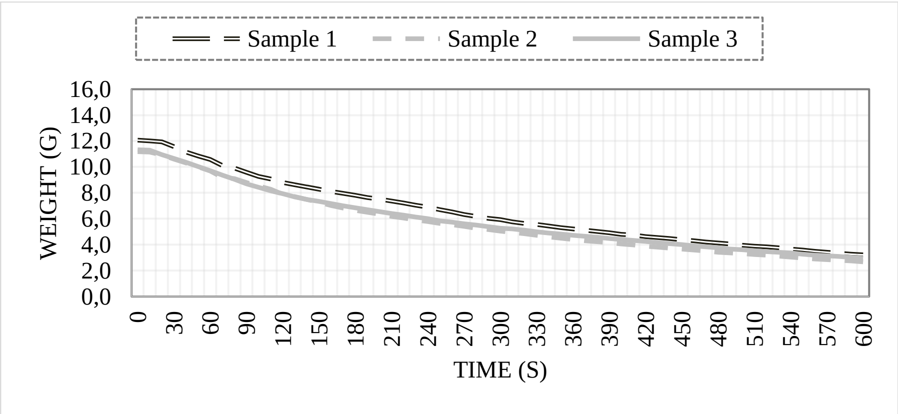

Fig. 1 The mass loss development of *Paulownia tomentosa* during thermal loading

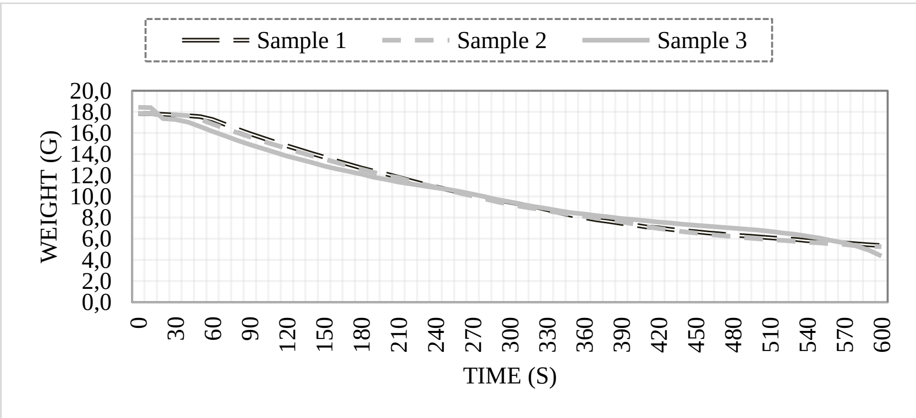

Fig. 2 The mass loss development of *Salix viminalis* variety Tora during thermal loading

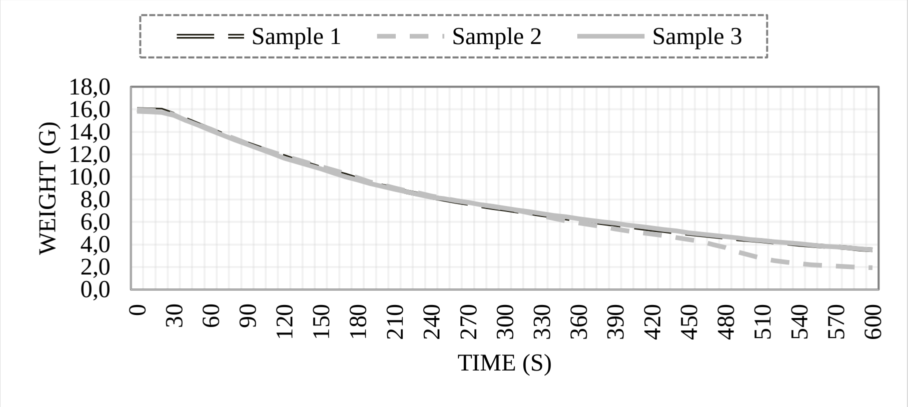

Fig. 3 The mass loss behaviour of *Populus x euroamericana* MAX 4 during thermal loading

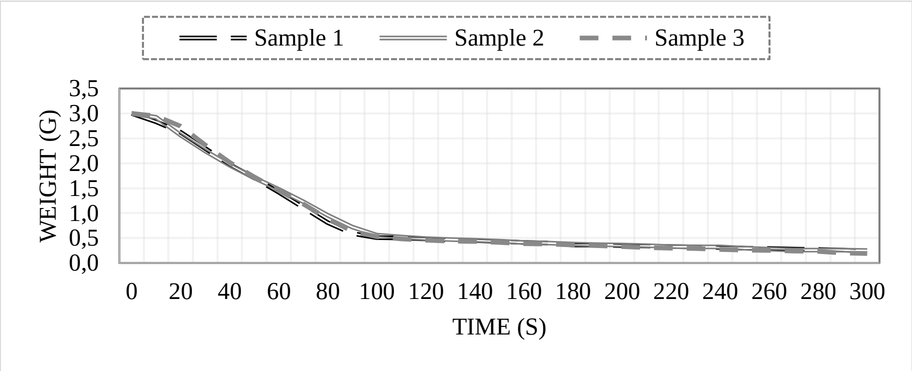

Fig. 4 The mass loss development of *Sida hermaphrodita* during thermal loading

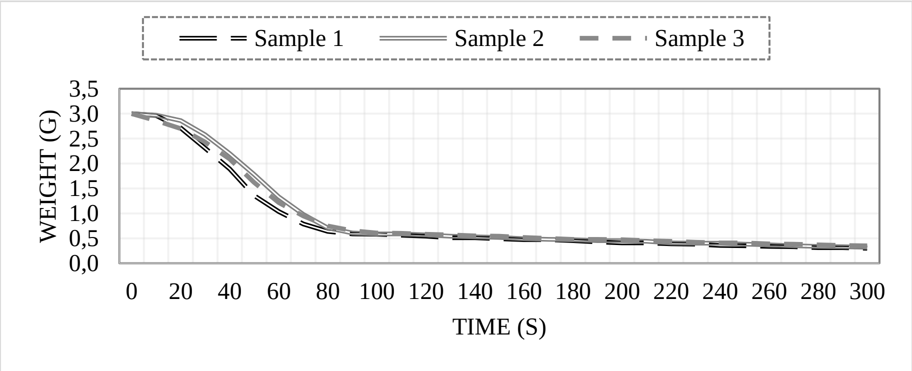

Fig. 5 The mass loss development of *Arundo donax* during thermal loading

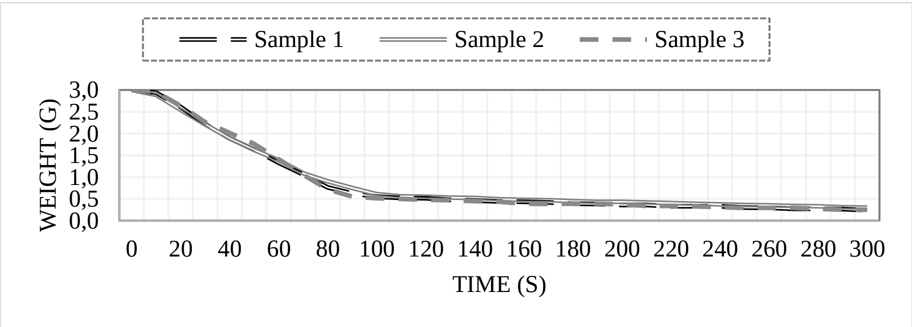

Fig. 6 The mass loss development of *Miscanthus x giganthus* during thermal loading

The differences in mass loss course of fast-growing tree species and energy crops are evident in the mass loss developments. The development of fast-growing-tree species mass loss is more linear, while the development of the energy crops showed the rapid mass loss in interval of 1-90 s. Then the mass loss rate decreased and became linear. The fact that in the first stage of thermal decomposition process the active pyrolysis occurred can be assumed. After that, only the passive pyrolysis process occurred.

In individual analyses of the samples, only small differences in the mass loss development of tested fast-growing tree and energy crop species samples can be distinguished.

Fig. 7 shows the differences in time of ignition and time of flame extinguishing.

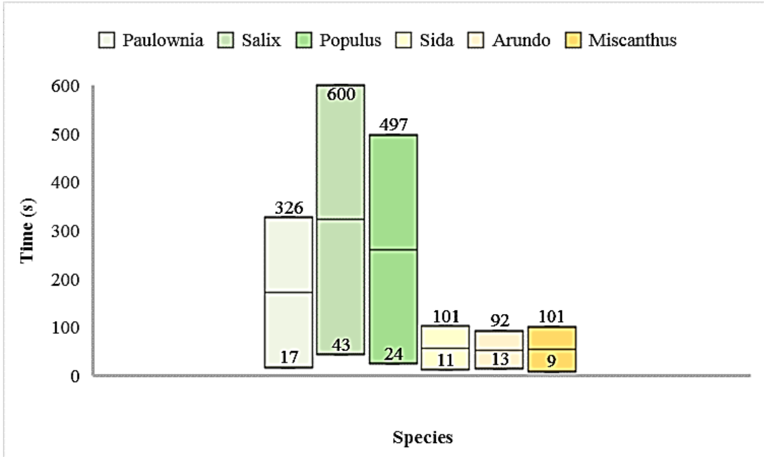

Fig. 7 Time of ignition and time of flame extinguishing of the tested samples

The results introduced in Fig. 7 showed significant differences in time of flame burning, when comparing the fast-growing tree species and energy crops. The energy crops reached much shorter time of flame burning. The results representing the mean length of flame burning of individual samples are as follows: *Paulownia tomentosa* of 309.33 s, *Salix viminalis* variety Tora of 557 s (however, the flames died out due to their manual extinguishing in 600 s), *Populus x euroamericana* variety MAX 4 of 473 s, *Sida hermaphrodita* of 90.33 s, *Arundo donax* of 79 s, *Miscanthus x giganthus* of 92.33 s.

DISCUSSION

The results of spontaneous ignition temperature examination showed that the samples were resistant to fire for a shorter time with increasing thermal loading (and higher spontaneous ignition temperature value). The results, in general, confirmed the fact that the values of induction time are decreasing with increasing values of temperatures. The very similar results were obtained also by ZACHAR *et al*. (2017) when testing Norway spruce and ThermoWood samples.

The activation energy of the *Populus sp.* cellulose was studied by LIANG *et al*. (2018). They applied the Kissinger-Akahira-Sunose method to determine it. The mean activation energy value of poplar in their experiments was calculated as 176.20 kJ·mol^{-1}. When compared to the activation energy calculated in this analysis, using the Arrhenius equation (145.60 kJ·mol^{-1}), the difference of -30.6 kJ·mol^{-1} was found.

MEIDI *et al*. (2010) studied the activation energy of two energy crops: *Arundo donax* and *Miscanthus x giganthus*, in the devotalisation and char oxidation steps. Activation energy for the *Arundo donax* was stated as 107.20 kJ·mol^{-1} and 96.40 kJ·mol^{-1} for *Miscanthus x giganthus*. Using the results of activation energy calculation for *Arundo donax* (144.30 kJ·mol^{-1}) introduced in this study, the difference of 37.1 kJ·mol^{-1} was found. Similar difference was found for the activation energy values of *Miscanthus x giganthus* (129.60 kJ·mol^{-1}), where the difference was of 33.20 kJ·mol^{-1}.

KOK AND ÖZGÜR (2013), who applied the Ozawa-Flynn-Wall, Kissinger and ASTM methods to calculate the activation energies of *Populus sp.* and *Miscanthus x giganthus* samples Quite different results achieved. Those methods require performing the TG/DTG and DSC analyses to calculate it. The activation energy values for Populus were calculated as follows: by the Ozawa-Flynn-Wall method it was of 219.20 kJ·mol^{-1}, by the Kissinger method 129.20 kJ·mol^{-1} and by the ASTM method it was of 138.1 kJ·mol^{-1}. The Miscanthus x giganthus activation energies: by the Ozawa-Flynn-Wall method was of 229.40 kJ·mol^{-1}, by the Kissinger method of 135.80 kJ·mol^{-1} and by the ASTM method was of 143.2 kJ·mol^{-1}.

The activation energies calculated by different methods show significant differences caused by application of different approaches to determination of thermal degradation process and different equations for stating the activation energy. The methods are still developing to find an approach which will be more suitable and precise and will eliminate the errors which present methods contain.

A non-standard method to estimate the mass loss of fast growing tree species and energy crops with the mass loss expressed in g·s^{-1}was applied. Other authors used the results of TG analysis expressed in mg.°C^{-1} or mg.°K^{-1}to determine it. Due to the incompatibility of the units we were not able to provide the comparison and discussion of those results.

CONCLUSIONS

The activation energy and mass loss of totally six species of fast-growing tree species and energy crops (*Paulownia tomentosa*, *Salix viminalis* variety Tora, *Populus x euroamericana* variety MAX 4 and *Sida hermaphrodita*, *Arundo donax*, *Miscanthus x giganthus*) is discussed in this paper. The results showed that the similar activation energy values were gatehered in all tested samples. They were in the range of 136.72 ± 7.28 kJ·mol^{-1}.

Studying the mass loss of the tested samples, we found the evident two-stage process of thermal degradation in case of energy crops, while the development of thermal degradation of fast-growing tree species was much more linear. When studying the mass loss, the time of autoigniton and time of flame extinguishing for each sample was recorded. The difference between the results of fast-growing tree species and energy crops was found. The energy crops flame burning lasted for less than 100 s, while the fast-growing tree species for more than 300 s. The *Salix viminalis* samples had to be extinguished manually after 600 s.

The spontaneous ignition temperature values of fast-growing tree species varied in the temperature range of 419.46 °C – 424.32 °C and the values of energy crop in the temperature range of 425.57 °C – 431.20 °C. The induction time values of the fast-growing tree species were in the range of 295.70 s – 329.90 s, while the energy crops in the range of 211.70 s – 231.50 s.

These results point out the energy crops to be more suitable for the use as a renewable energy source, not onlyin terms of the energy properties, but especially due to higher yields in comparison to fast-growing tree species. The annual yields of fast-growing tree species like *Salix sp*. and *Populus sp*. in production years vary from 10 to 18 t·ha^{-1}·year^{-1} (CHUDIKOVA *et al.*, 2010), while the *Sida hermaphrodita* ranges 12-20 t·ha^{-1}·year^{-1}, *Miscanthus x giganthus* of 15-30 $t \cdot ha^{-1} \cdot year^{-1}$ and *Arundo donax* even of 63 $t \cdot ha^{-1} \cdot year^{-1}$ (Ekocentrum, 2014).

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