

Analysis of Spontaneous Ignition Temperature and Flash Point for Predicting Fire Risk in Biomass Fuel Pellet Storage

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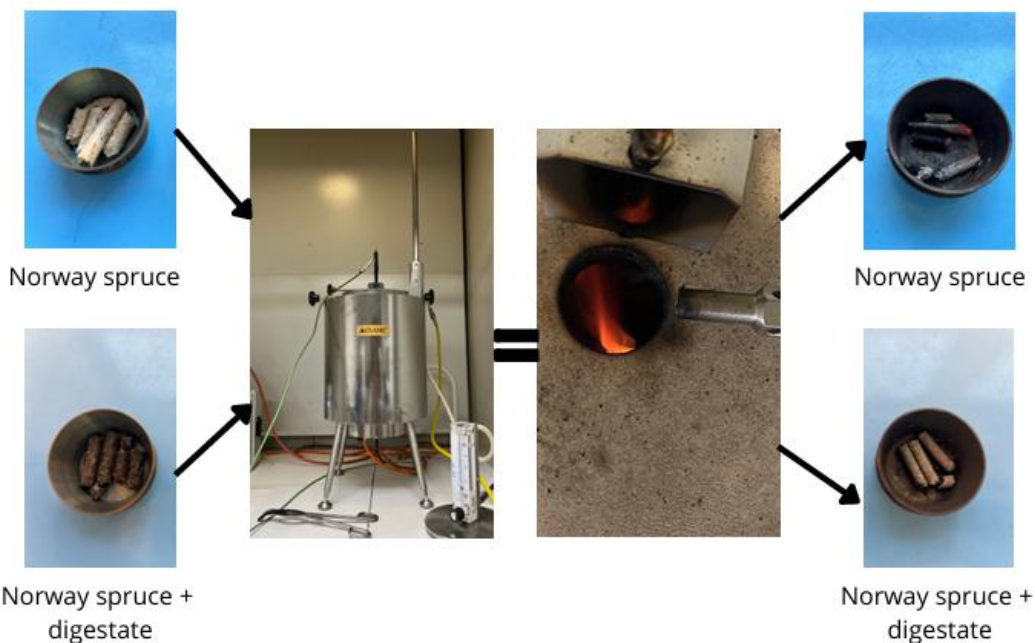
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GRAPHICAL ABSTRACT

Wood species	Activation energy of spontaneous ignition temperature (KJ.mol ⁻¹)	Activation energy of flash point temperature (KJ.mol ⁻¹)
Norway spruce	65.42	37.06
Norway spruce + digestate	51.50	42.06

Example before burning



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Biomass pellet fuel is one of the alternative renewable energy sources, which has an important place in the fuel market. This article analyses the spontaneous ignition temperature and flash point temperature, which are the most important characteristics for fire risk prediction, especially in the storage. The Norway spruce pellet and experimental pellet with digestate sawdust were tested according to ISO 871: 2010. The novelty of this research is the application of the Setchin Furnace test. The spontaneous ignition temperature for the Norway spruce pellet and the experimental pellet were established as 420 and 450 °C, respectively. The flash point temperature for the Norway spruce pellet was 330 °C and for the experimental pellet 320 °C. The activation energy was higher for the Norway spruce pellet (65.4 KJ.mol⁻¹) for spontaneous ignition temperature and for the experimental pellet (42.1 KJ.mol⁻¹) for flash point temperature.

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Keywords: Spontaneous ignition temperature; Flash point temperature; Setchin Furnace test; Wood pellets

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INTRODUCTION

Biomass-based fuels, such as wood pellets, have emerged as a sustainable and renewable energy source in recent decades, offering an alternative to fossil fuels for mitigating climate change. One of the most commonly used materials for pelletization is Norway spruce (*Picea abies*) sawdust, due to its favorable combustion properties and widespread availability. However, the integration of supplementary materials, such as digestate from biogas production, into pellets is increasingly being explored to enhance waste valorization and resource efficiency. The use of such digestate has been analyzed by Czekala (2021). Studies such as those conducted by Kratzeisen *et al.* (2010), have examined the applicability of digestate from biogas plants as a solid biomass fuel and evaluated its classification based on existing regulations.

Testing materials and their behavior under the influence of heat has always played a crucial role, whether dealing with organic or inorganic materials. The need for such testing is essential to determine their properties under various conditions that may arise. The findings from these tests are then applied in different ways. For instance, testing is critical to assess the suitability of fire retardants for specific materials (Terzi *et al.* 2011) or the appropriateness of protective equipment (Kubás *et al.* 2024). In the case of wood, these tests are also important for modeling large-scale forest fires. Understanding the

behavior of such natural events is crucial for developing models and predictions that directly impact the protection of human lives (Titko *et al.* 2021).

Different types of wood species vary in their properties. In fire engineering, properties that affect wood behavior during combustion are of particular interest. Significant properties include ignition temperature, flash point, and activation energy. This research focuses on determining these physical properties. Ignition temperature can be defined as the minimum temperature to which the air must be heated so that the sample put in the heated air environment ignites (Harangozó *et al.* 2024).

The importance of testing is emphasized by Chumakova (2019), who stated that the most important factor determining the fire danger of wood is its susceptibility to the emergence and development of combustion when heated in air. The importance was also highlighted by Zhai *et al.* (2017), who stated that when heated, the surface and inner temperature of fuel rises, leading to pyrolyzing, release of combustible gas, and finally combustion while the critical conditions for ignition are met.

Several studies have focused on testing specific types of wood using the Setchkin furnace, including (Chrebet *et al.* 2013; Horváth *et al.* 2014; Martinka and Chrebet 2014; Zachar *et al.* 2017; Majlingová *et al.* 2018; Čabalová *et al.* 2019). Comprehensive measurements and comparisons of the activation energy of wood dust, solid wood, and pellets made from Norway spruce were conducted by Martinka *et al.* (2015). They found that, in comparison, the pellets exhibited the highest spontaneous ignition temperature, reaching 460 degrees Celsius. Consequently, this also resulted in the greatest activation energy requirement ($59 \text{ kJ}\cdot\text{mol}^{-1}$). The activation energy of pine and beech wood was determined also in the work of Janković (2014). The thermal decomposition of spruce pellets was also investigated by others (Mar'yandyshev *et al.* 2015; Ondro *et al.* 2018; Vitázek *et al.* 2019; Tian and Perré 2023).

Isothermal phases and the decomposition of beech and spruce wood were examined by Preimesberger *et al.* (2024), who found that the decomposition of hemicelluloses and cellulose peaks at around 360 °C. Vermesi *et al.* (2017) studied spontaneous ignition of wood subjected to transient irradiation and identify strengths and gaps in the topic. Zachar *et al.* (2012) tested various parts of spruce (trunk, branch, and root) and determined both the spontaneous ignition temperature and the flash ignition temperature of these parts. Spruce pellets were subjected to comprehensive testing and fire risk evaluation by Martinka *et al.* (2014). Van Blijderveen *et al.* (2010) presented the effects of primary air flow velocity, particle size, moisture content, and the addition of inert material to the fuel bed on the spontaneous ignition behavior of wood fuel.

This study aimed to investigate the reactivity characteristics of standard wood pellets produced from Norway spruce sawdust and an experimental pellet containing a 50:50 mixture of Norway spruce sawdust and digestate. The flash point and spontaneous ignition temperatures of the samples were determined in accordance with ISO 871: 2023. Additionally, the activation energy of combustion was calculated to provide further insights into the ignition and thermal decomposition behavior of the pellets.

By comparing the standard and experimental pellets, this research seeks to contribute to the understanding of how digestate incorporation influences the combustion properties of biomass fuels.

EXPERIMENTAL

One species of commonly available wood pellets and one experimental sample were selected, and they were provided directly by the producer (Fig. 1). The standard pellets were produced from Norway spruce sawdust. The experimental sample consisted of Norway spruce and digestate sawdust in proportions 50% to 50%. The digestate refers to the residual material left after the anaerobic digestion of biodegradable feedstocks. It mainly comprises water, organic compounds that were not fully broken down during fermentation, minerals, and the biomass of microorganisms (Czekala *et al.* 2023). The quantity and composition of the digestate can vary significantly, with the type of substrate used in the anaerobic digestion process being one of the most influential factors (Czekala *et al.* 2023). Digestate consists of two parts – the liquid phase (filtrate), which is used as an organic fertilizer, and the solid phase (separate), which can be used as an energy source or as bedding for livestock (Bialowiec *et al.* 2015). Experimental pellet samples were obtained from a company in Slovakia specializing in pellet production. Due to ongoing tests, the company did not agree to provide detailed chemical analyses. The digestate used in the pellets originated from plant production (source: corn silage, manure, and slurry).

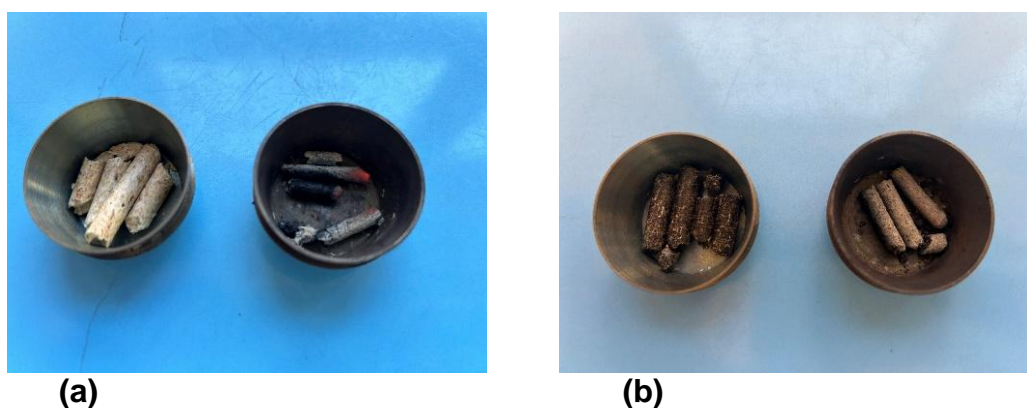


Fig. 1. Sample of pellets before and after the experiment: (a) Norway spruce pellet; (b) Experimental pellet

The mass of the sample was 3.0 ± 0.2 g. It was conditioned at 23 ± 2 °C and a relative humidity of 50 ± 5 % for 40 h, according to STN EN ISO 291. The basic parameters of pellets are listed in Table 1. The experimental sample had a higher moisture content as it contained 50% digestate. According to other studies, the moisture content of the digestate itself ranges from 6.4% to 8.1% (Tlustoš *et al.* 2014). The present results are within the given moisture range.

Table 1. Basic Parameters of Samples

Wood species	Class	Country	Moisture w (%)
Norway spruce	A1	Slovakia	5.8
Norway spruce + digestate	Experimental sample	Slovakia	6.6

First, the spontaneous ignition temperature and flash point temperature were determined according to the STN ISO 871: 2023. The standard test procedure was based on the minimum temperature at which the sample was ignited. In a hot-air furnace (Fig. 2), the sample was heated at different temperatures with the application of the ignition

flame (flash point temperature) or without (spontaneous ignition temperature). The temperatures were measured by the data logger Pico technology USB TC – 08.

The sample was inserted into the hot-air furnace and observed for 10 min. After 10 min, the temperature was reduced or raised to 50 °C, depending on whether the sample was ignited. The experiment was applied to the new sample. After the temperature range was established, the test started at the temperature of 10 °C lower than the highest temperature within the temperature range and continued with reducing the temperature by 10 °C to the temperature at which the sample did not ignition for 10 min. The test procedure was the same when the spontaneous ignition temperature or flash point temperature was established. The spontaneous temperature was recorded at the lower temperature. During that 10 min period, observations were made of the burning or glowing of the sample. The flash point temperature was recorded as the lower temperature at which, during the 10 min, the sample was undergoing ignition (STN ISO 871: 2023).

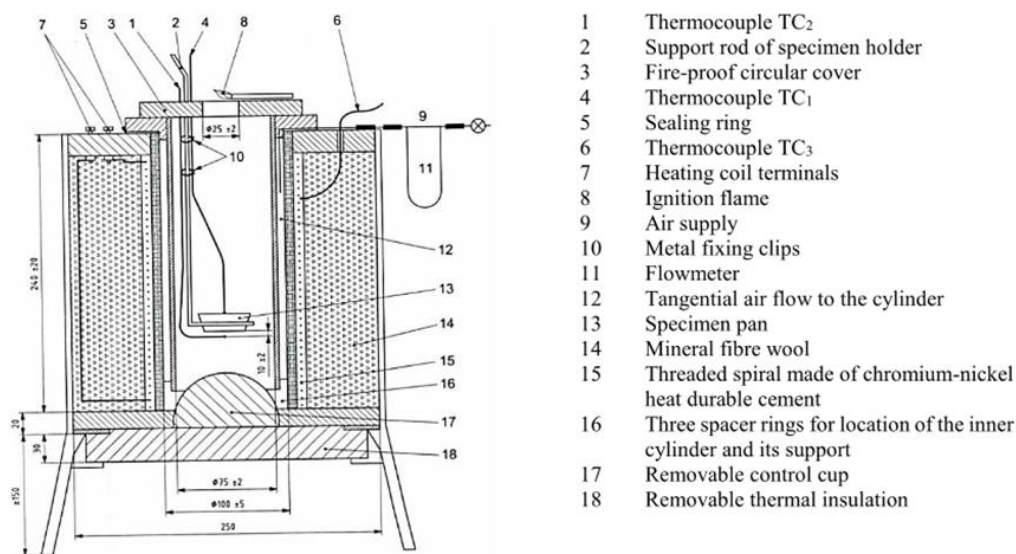


Fig. 2. The scheme of the hot-air furnace (According: STN ISO 871)

A notable feature of this work is the application of the Setchin Furnace test, *i.e.*, the thermoanalytical methods by Martinka *et al.* (2015) and Zachar *et al.* (2017). After determining the minimum spontaneous / flash point temperature, the temperature was gradually increased by 10, 20, 30, 40, 50, 60, 70, 80, 90, and 100 °C, and the ignition time was measured. Each test was repeated three times, and the results of the ignition time and spontaneous / flash point temperature are the average values.

The exponential equations for dependence between the ignition time and the inverse values of thermodynamic temperature were derived in Microsoft Excel software. The activation energy was calculated according to the equation published by Semenov (1959). The activation energy is an analogy to the Arrhenius equation, as follows,

$$E = \ln \frac{\tau}{A} \times R \times T \quad (1)$$

where E is the activation energy ($\text{J}\cdot\text{mol}^{-1}$), τ is the ignition time (s), A is the pre-exponential factor (-), which are calculated from the exponential equation, R is the universal gas constant ($8.314 \text{ J}\cdot\text{K}^{-1}\cdot\text{mol}^{-1}$), and T is ignition thermodynamic temperature (K).

RESULTS AND DISCUSSION

The spruce and experimental pellets showed higher ignition temperatures for the experimental sample (450 °C) and flashpoint temperatures for spruce pellets (330 °C, Fig. 3). These differences were attributed to the presence of digestate in the experimental sample at a 50% level. In the literature, data show the ignition temperature of biogas in the range 650 to 750 °C, during the production of which a waste product in the form of digestate is generated. Jad'ud'ová *et al.* (2023) tested the ignition temperature of the solid phase of digestate. The initial burning process began at a temperature of 206 °C with a sample thickness of approximately 1 cm. The Catalog of fire-technical properties of materials reports an ignition temperature of 390 to 400 °C and a flashpoint temperature of 350 to 360 °C for Norway spruce wood (Ministry of Interior of the Slovak Republic 1984). Variability in the results was observed during the induction period. Experimental samples required comparatively less time at the ignition temperature (207 s) as well as at the flashpoint temperature (389 s) than spruce samples (Fig. 3).

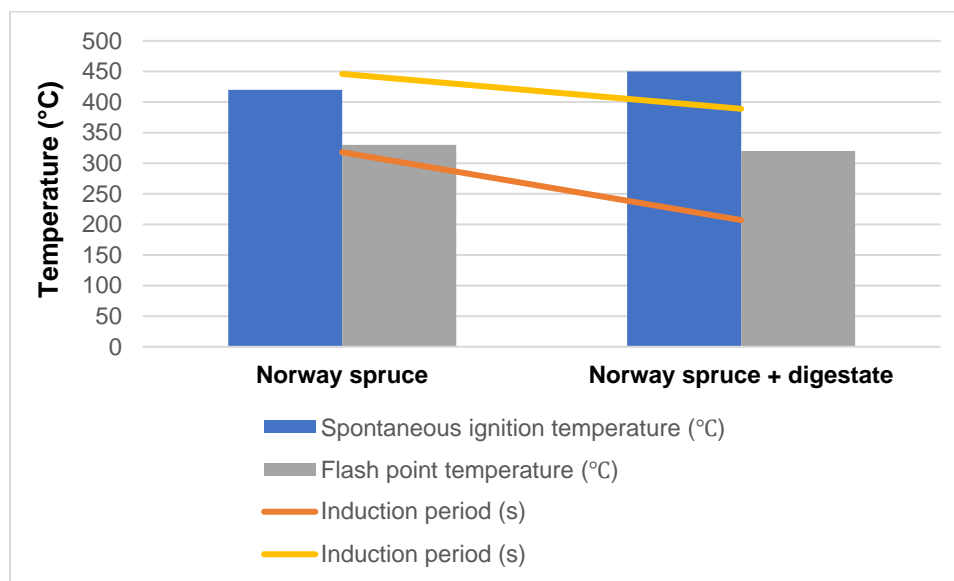


Fig. 3. Graphical comparison of the temperature and time of ignition and flare-up of the tested pellets

Table 2 presents the average values (from three measurements) of the lowest determined ignition temperature, flashpoint temperature, induction period, and mass loss. One of the accompanying phenomena of thermal degradation of materials is mass loss. During the determination of both ignition and flashpoint temperatures, pellets of Norway spruce lost more of their original mass compared to the experimental samples. In both tests, pellets of Norway spruce retained only one-third of their original mass. Experimental samples retained less than half of their mass (41%) during the determination of the flashpoint temperature.

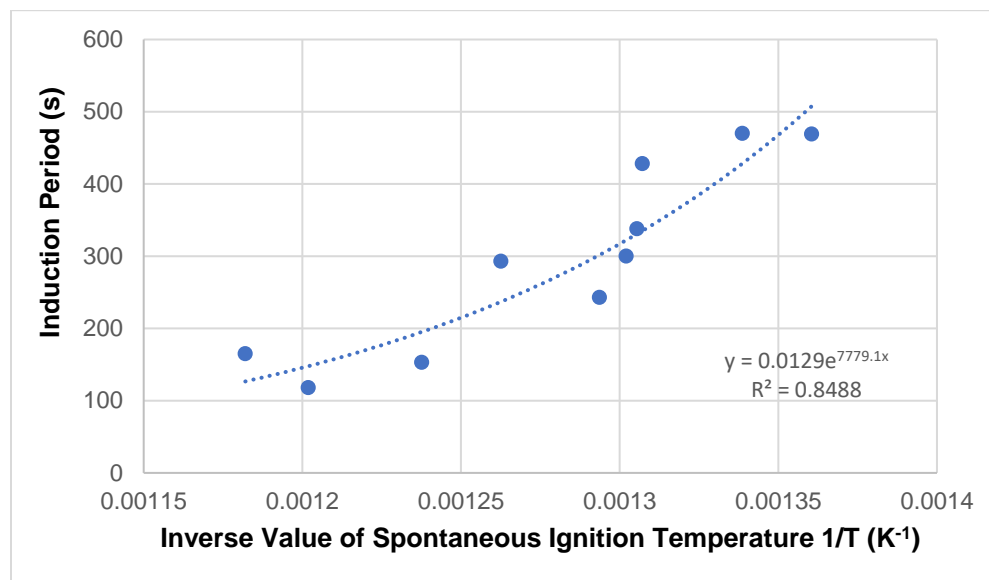
Mitterová *et al.* (2016) investigated the mass loss of briquettes made from Norway spruce sawdust and concluded that during the determination of the flashpoint temperature, they lost half of their mass, which was 30% less compared to the mass loss during ignition. In the present experiment, pellets of Norway spruce lost more mass, which can be attributed to the differing test temperatures.

Table 2. The Results of Spontaneous Ignition Temperature and Flash Point Temperature of Pellets

Wood species	Spontaneous ignition temperature (°C)	Induction period (s)	Mass low (%)	Flash point temperature (°C)	Induction period (s)	Mass low (%)
Norway spruce	420	318	82	330	446	70
Norway spruce + digestate	450	207	73	320	389	59

After determining the minimum ignition and flashpoint temperatures, the Setchin Furnace test was conducted. Based on the test results, exponential equations were formulated, and the correlation coefficient was calculated. For the ignition temperature, the minimum temperature for pellets of Norway spruce was determined during the first measurement (462 °C) at 469 seconds from the start of the test. During the test, the values of the induction period decreased with increasing temperature.

An anomaly occurred during the fourth measurement (243 seconds at 500 °C), which could have been caused by the pressing of the pellets and their breaking during the weighing of the required sample mass (3 g). The minimum time for the ignition temperature of pellets of Norway spruce was achieved during the penultimate measurement (118 seconds) at a temperature of 559 °C. The pellets of Norway spruce achieved a correlation value of 0.8488, indicating a strong dependency between temperature and time (Fig. 4).

**Fig. 4.** Graph of dependency between the inverse value of spontaneous ignition temperature and induction period of Norway spruce pellets

The experimental pellet sample in the modified ignition test exhibited a fluctuating temperature profile, as shown in Fig. 5. The addition of digestate at a 50% to 50% ratio caused instability in the sample. In the regression curve, the point at 240 seconds represented the initial time at which self-ignition of the experimental sample occurred at a Setchin furnace temperature of 450 °C. A visible time drop (174 seconds) was recorded at a Setchin furnace temperature of 460 °C, even during repeated measurements. Temperature

fluctuations were particularly noticeable at lower temperatures, specifically during the fourth and fifth measurements, with the hot-air furnace set to 480 and 490 °C. The self-ignition process of the experimental sample at lower temperatures showed irregular behavior. Since the chemical composition of the sample is not available, it can be assumed that this was caused by the presence of digestate in general.

The minimum temperature for the experimental pellet sample was determined during the fifth measurement (425 °C) at 143 seconds from the start of the test. The minimum ignition time was recorded during the final measurement (100 seconds) at a temperature of 465 °C. The exponential equation indicated a weak correlation between temperature and time ($r = 0.1634$; Fig. 5), which can be regarded as a negligible effect.

Using the modified Setchin Furnace test, the lowest flashpoint temperature for pellets of Norway spruce and experimental pellets was determined. For pellets of Norway spruce, the lowest flashpoint temperature was recorded during the second measurement, with a value of 343 °C at 399 seconds. A nearly identical result was obtained during the first measurement (344°C), but with a significantly longer time of 461 seconds from the start of the test. In both tests, the sample burned with a flame. The lowest flashpoint temperature for the experimental sample was recorded at 326 °C after 390 seconds. The shortest time (165 seconds) was recorded for the experimental pellets at a temperature of 373 °C. The maximum flashpoint temperature was measured during the ninth measurement (379 °C) at a time of 215 seconds.

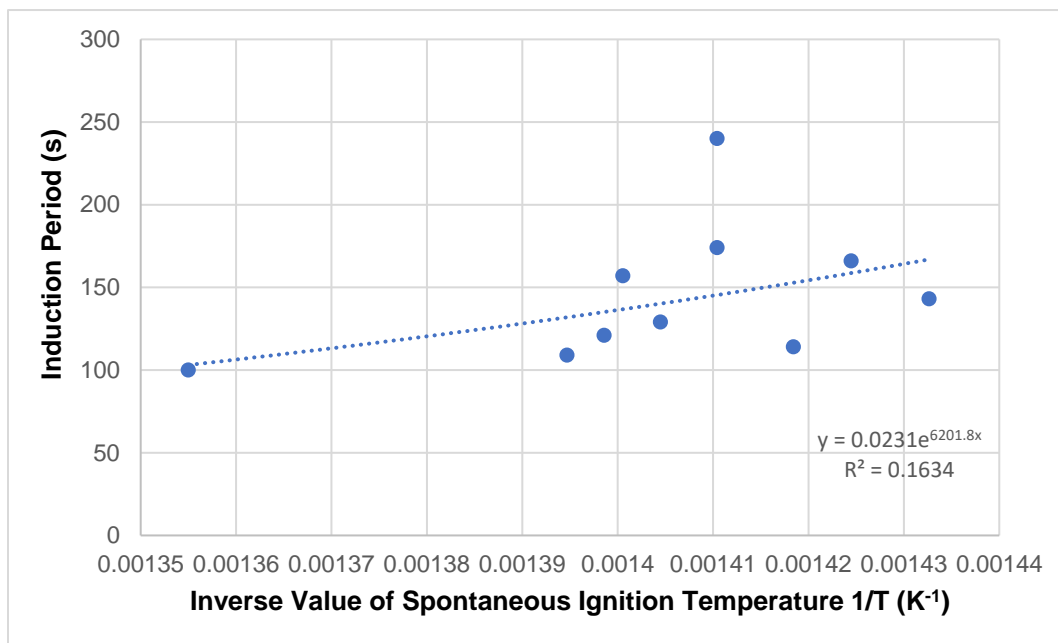


Fig. 5. Graph of dependency between the inverse value of spontaneous ignition temperature and induction period of Experimental sample of pellets

Specimens prepared from Norway spruce pellets and experimental pellets showed a very high correlation between the flashpoint induction period and the inverse value of the thermodynamic temperature, with correlation coefficients of $r = 0.9205$ (Fig. 6) for Norway spruce pellets and $r = 0.8123$ for experimental pellets (Fig. 7).

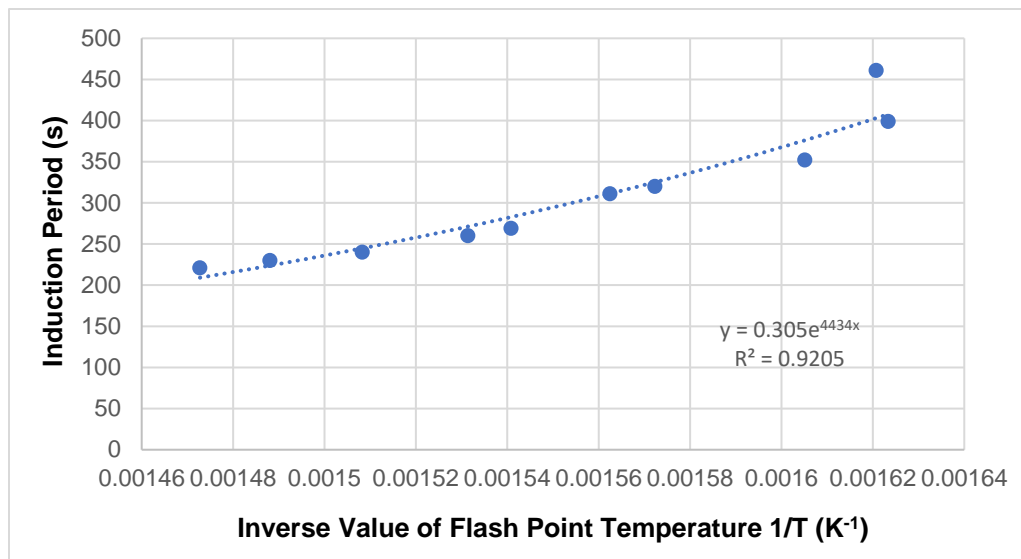


Fig. 6. Graph of dependency between the inverse value of flash point temperature and induction period of Norway spruce pellets

As shown in Figs. 6 and 7, the studied samples exhibited a linear curve progression. Measurements for the Norway spruce pellet samples oscillated around the trendline with a standard temperature pattern. The experimental sample of pellets displayed a fluctuating temperature profile even during the flashpoint test (Fig. 7).

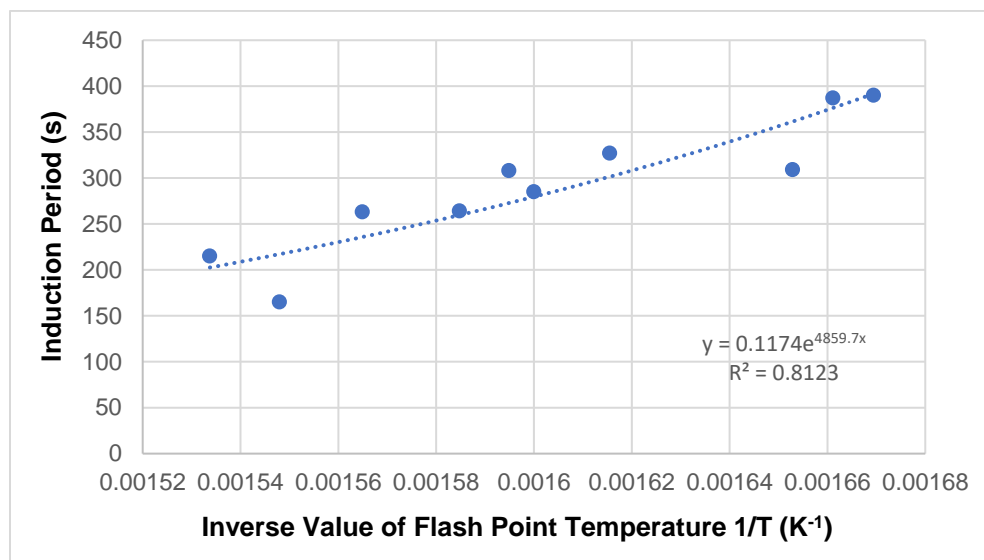


Fig. 7. Graph of dependency between the inverse value of flash point temperature and induction period of Experimental sample of pellets

The activation energy parameter is more suitable for comparing the studied samples, as the procedure according to ISO 871:2010 allows for determining ignition and flashpoint temperatures with an accuracy of 10 °C. Measuring the fundamental input variable for calculating activation energy can be done with an accuracy of 1 second (Martinka *et al.* 2015). The calculation of activation energy shows that Norway spruce pellet samples had a higher activation energy for ignition (65.42 KJ·mol⁻¹) compared to

flashpoint ($37.06 \text{ KJ}\cdot\text{mol}^{-1}$, Table 3). The experimental pellet samples exhibited significantly lower activation energy for self-ignition due to the 50% share of digestate sawdust, which resists higher temperatures. However, the activation energy for the flashpoint was higher in the experimental samples ($42.06 \text{ KJ}\cdot\text{mol}^{-1}$) compared to Norway spruce pellets. It can be assumed that the experimental sample pressed with digestate sawdust is not a homogeneous material, and therefore variability in the ignition temperature of a few degrees Celsius should be expected.

Table 3. Activation Energy for Norway Spruce Pellets and Experimental Sample of Pellets

Wood species	Activation energy of spontaneous ignition temperature ($\text{KJ}\cdot\text{mol}^{-1}$)	Activation energy of flash point temperature ($\text{KJ}\cdot\text{mol}^{-1}$)
Norway spruce	65.42	37.06
Norway spruce + digestate	51.50	42.06

Pellets of Norway spruce were analyzed by Martinka *et al.* (2015), and they reported an activation energy of $59.0 \text{ KJ}\cdot\text{mol}^{-1}$ for spontaneous ignition. The activation energy was higher in the present work ($65.4 \text{ KJ}\cdot\text{mol}^{-1}$), which was attributed to the fact that the pellet is not a homogenous material, and their pressing can affect the results. The Norway spruce wood is the most frequently used material to test fire characteristics, such as spontaneous ignition temperature or flash point temperature. Zachar *et al.* (2017) established the activation energy of spontaneous ignition temperature for Norway spruce wood at $67.2 \text{ KJ}\cdot\text{mol}^{-1}$ and the activation energy at the flash point temperature as $39.1 \text{ KJ}\cdot\text{mol}^{-1}$. Their results are comparable to the present results. Matejova (2012) tested a set of specimens of Norway spruce wood, and the activation energy of spontaneous ignition temperature was $62.0 \text{ KJ}\cdot\text{mol}^{-1}$ and the activation energy at the flash point temperature was $28.5 \text{ KJ}\cdot\text{mol}^{-1}$. Veľas *et al.* (2019) tested Norway spruce in the solid form and their calculation of activation energy of spontaneous ignition was $57.6 \text{ KJ}\cdot\text{mol}^{-1}$. These results are lower than the present ones, but the wood is not a homogeneous material. Thus it is expected that the ignition temperature will vary by a few degrees Celsius.

LIMITATIONS AND CONTINUATION OF THE RESEARCH

Existing studies on this topic have primarily focused on standard pellets and their energy potential. The present article has analyzed the spontaneous ignition temperature and flash point temperature as standard pellets (from Norway spruce) and experimental pellets (from Norway spruce and digestate). The field of digestate fire safety has not been sufficiently researched. These parameters are important storage in terms of fire risk. The new aspect of the article is the application of the Setchin Furnace test and the use the pellets from digestate.

Firstly, the present article analyzed only two fire risk parameters (self-ignition temperature and flash point temperature). The Setchin Furnace test itself can be modified not only in terms of temperature but also by changes in mass, or by monitoring mass loss over time. In the future, it is planned to expand the research to include the analysis of combustion emissions. Secondly, the experimental samples had a 50% Norway spruce and

50% digestate ratio. A potential avenue for further research is to vary the digestate proportion in the pellet sample and test the ignition and flash point temperatures.

CONCLUSIONS

This study examined the thermal properties and combustion behavior of standard Norway spruce wood and experimental pellets made from a 50:50 mixture of Norway spruce and digestate. The analysis focused on key parameters, including flash point, spontaneous ignition temperature, activation energy, and the correlation coefficient between time and temperature.

1. The pellets prepared with 50% of digestate demonstrated a surprising result: they required lower activation energy at the spontaneous ignition temperature ($51.5 \text{ kJ}\cdot\text{mol}^{-1}$) compared to spruce wood ($62.4 \text{ kJ}\cdot\text{mol}^{-1}$), suggesting that the inclusion of digestate significantly influenced the ignition characteristics. The activation energy at the flash point temperature was higher in the case of the experimental pellet ($42.1 \text{ kJ}\cdot\text{mol}^{-1}$) in comparison to spruce wood ($37.1 \text{ kJ}\cdot\text{mol}^{-1}$).
2. In terms of spontaneous ignition temperature, the experimental pellet achieved a value of $450 \text{ }^\circ\text{C}$, compared to $420 \text{ }^\circ\text{C}$ for spruce wood, while the flash point for the pellet was 320 and $330 \text{ }^\circ\text{C}$ for spruce wood.
3. The correlation coefficient between time and temperature was determined to be 0.8488 for the spontaneous ignition temperature and 0.9205 for the flash point temperature of spruce wood, indicating higher dependence of the experimental pellet ($r = 0.1634$ for spontaneous ignition temperature, and $r = 0.8123$ for flash point temperature).
4. These findings highlight that incorporating digestate into wood pellets affects the combustion properties, with potential implications for the use of alternative biomass fuels. Further research is needed to explore the mechanisms behind these differences and assess their impact on practical applications in energy systems.

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