

## Influence of temperature and duration of pyrolysis process on calorific value of cereal straw biochar and dust explosive index

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**Abstract.** The challenges of the modern world largely concern the energy sector and determine the search for new environmentally friendly and, above all, renewable energy sources. We can consider biochar or materials obtained by pyrolysis as an alternative and green fuel. Substrates for the production of such fuels can be biomass of agricultural origin, including straw. In the present study, wheat straw, oat straw, and triticale straw were used to produce pyrolysates. This paper evaluates the modification of pyrolysis parameters by assessing selected properties of pyrolysates, focusing on the calorific value and parameters characterizing the dust explosion hazard. There was an average increase of 30% in the calorific value of all biocarbons prepared from the three types of straw in relation to the control sample. The anaerobic thermal treatment resulted in a maximum increase in the calorific value to 26.43 MJ kg<sup>-1</sup>. An increase in such parameters as dust explosive index, the maximum rate of pressure buildup and the maximum explosion pressure was found. However, there was no effect on the increase in the dust explosion risk. Noteworthy, the results indicate that it is possible to use various types of straw to produce biochar fuels by anaerobic thermal treatment without increasing the probability of dust explosion.

**Keywords:** straw, biochar, pyrolysis, dust explosion rate, biofuels

### 1. INTRODUCTION

With the constant technological development and the world's limited fossil fuel resources, we are forced to look for new ways to obtain energy. Until some time ago, the

problem had mainly been of interest to scientists, but when such problems as atmospheric pollution, climate change on Earth, increased consumption, and dwindling drinking water supplies began to increasingly affect the planet's population, discussions began in wider circles. An ideal solution seems to be renewable energy sources, which, unlike conventional sources, are not in short supply, as their resources are able to renew in a relatively short time. Additionally, their negative impact on the environment is small and can be reduced to the necessary minimum. Biomass is one of the main branches of renewable energy sources. Other leading sectors of renewable energy sources are solar, water, and wind energy, especially offshore wind farms. It should also be noted that photovoltaics is currently developing very fast. The number of energy prosumers is growing very dynamically. Among the many types of biomass, straw is used as a substrate for energy production, as its produced surplus, not used for food, can be processed into biofuel and used for energy purposes. Straw is a very good substrate for many matter-to-energy conversion processes. In addition to the most popular thermal processing of biomass, such as incineration, co-firing, or biogas production, pyrolysis is also a viable energy option. The possibility of using biomass, including straw, as an energy carrier is guaranteed by its structure and composition. Biomass can be

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characterized as a composite material in which the main component is cellulose, which makes up about 50% of its chemical composition. Cellulose is a polysaccharide made up of many molecules and occurring in a crystalline form (2/3 of the content) and in a smaller amount (30%) in an amorphous form (Burczyk, 2011). The other half consists of hemicellulose (~25%), a polysaccharide made up of such compounds as glucose and arabinose, and lignin (~25%), which provides plant cells with compactness and flexibility (Maga *et al.*, 2010). In addition, the biomass composition is supplemented by water and trace amounts of extractives and inorganic compounds. The moisture content in biomass is important for heat transfer processes as well as for transport and storage of the material (Harasim, 2011). Traditionally, as in the rest of the world, straw in Poland was used primarily in agriculture. Nowadays, as a result of modernization of farms and growth of grain crops, there is a growing surplus of straw in Poland. In 2019, Poland produced 19,585.5 thousand tons of straw from basic cereals, namely rye, oats, barley, wheat, millet, and triticale, in a total area of 6 million hectares (Statistics Poland 2020). Calculating the surplus of straw that can be used as biofuel, it can be concluded that the amount of energy that can be produced from this plant material is 92.1 PJ/year (Igliński *et al.*, 2019). For the energy industry purposes, the most common agricultural waste can be divided into two categories. One is yellow straw (freshly harvested straw) and the other is called gray straw (weathered). The “yellow” straw is characterized by relatively high moisture content (20-30%); hence, it has a lower calorific value (Owczuk and Kołodziejczak, 2011). An additional disadvantage of this type of straw is its content of alkali metals and chlorine compounds, which can be catalysts for corrosive processes in boilers and installations in which this fuel is burned. “Gray” straw is characterized by significantly lower moisture content (< 10%). This type of straw is most useful in the energy industry. Gray straw contains significantly less chlorine, sulfur, and potassium compounds which reduces the corrosive processes in the boiler. The disadvantages of this type of straw include its brittleness, making harvesting efficiency low, and harvest losses that can reach 20-30% in this case (Karcz *et al.*, 2013). Straw intended to be used as a fuel for energy production should be characterized by appropriate parameters: moisture content (12-22% for dry straw), calorific value (from 14 to 16 MJ kg<sup>-1</sup>, depending on the type of grain), and content of such elements as carbon and sulfur (Owczuk and Kołodziejczak, 2011). The pace of decomposition and decay is also an important factor, which affects the content of carbon and hydrogen compounds in the straw (Lewandowski and Ryms, 2013).

Based on the method of preparation, the form of compression, and the purpose of biofuel made from straw, it can be divided into bales, briquettes, and pellets. Every biomass conversion method deals with chemical conversion, as the chemical energy in biomass is used to produce

energy. Chemical reactions occur as a result of biofuel-to-energy conversion technologies, such as combustion, co-firing, gasification, and fermentation as well as all other methods. For the most part, these are oxidation reactions, since biomass contains large amounts of hydrogen and carbon reductants. Since biomass has a different chemical composition than coal, the installation should be designed taking into consideration the difference in the composition of flue gases and ashes of the two fuels. The presence of alkali metals in biomass, which have the ability to pass into the gas phase, as well as the chlorine and sulfur content of coal, changes the chemistry of the combustion process compared to the thermal conversion of these materials in separate plants (Karami *et al.*, 2021; Lewandowski, 2012; Lewandowski *et al.*, 2010; Lewandowski and Ryms, 2013).

Plant biomass, including straw, can be refined through pyrolysis, *i.e.* the thermochemical conversion of organic matter into energy in anaerobic conditions (Lewandowski and Ryms, 2013). Pyrolytic processes are classified as intermediate transformation ones. This means that the physical and chemical reactions that occur during the course of the process change the energy of the fuel used into another form. Such a method of biomass conversion involves many chemical reactions (Kufka and Poterała, 2015). As a biomass conversion process, pyrolysis can be broadly divided into two main stages. In the first stage, there are primary reactions, *i.e.* degassing of the material, as well as thermal decomposition of the main components of the matter. During this stage, such reactions as dehydration, dehydrogenation, isomerization, and decarboxylation take place. The second stage involves secondary reactions in the matter. These are condensation and polymerization (Retajczyk and Wróblewska, 2018). The process of thermal decomposition of biomass through pyrolysis leads to the formation of three main end products, divided according to their physical state: solid: biochar (also known as carbonisate), which contains sizable amounts of elemental carbon and organic compounds and solid particles not converted in the process, gaseous: pyrolytic gas, which is a mixture of combustible gases, such as H<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub>, water vapor, gaseous hydrocarbons, and liquid vapors, and liquid: oil and tar, which are a mixed form of phenols, hydrocarbons, and various organic compounds (Klein *et al.*, 2012). Pyrolysis can be carried out at different temperatures. When the implementation scope of the process is from 450 to 700°C, it is then referred to as low-temperature pyrolysis. The pyrolysis process taking place at temperatures in the range of 900-1200°C is called high-temperature pyrolysis or coking (Nadziakiewicz *et al.*, 2012). The other factor according to which pyrolytic processes are classified is the speed of the process. For this purpose, a distinction is made between fast pyrolysis (the heating time of the raw material in the reactor is 0.5 s in the case of instant pyrolysis) and slow pyrolysis (the material can stay in the reactor even up to several minutes) (De Wild *et al.*, 2011).

In the case of slow pyrolysis carried out at a relatively low temperature (400-500°C) with the time of stay of the raw material in the reactor from a few to as much as thirty minutes, the main product will be a solid fraction in the form of biochar. There is also an intermediate type of the pyrolysis process - moderate pyrolysis. It is conducted in the temperature range of 500-600°C, and the time of stay of the substrate in the pyrolytic chamber is 10-20 s. The products of this process also include biochar, bio-oil, and gas, but the largest percentage of the final material is liquid (Malińska and Dach, 2015).

Considering the numerous processes involved in converting plant biomass into the form of solid biofuels and biochar fuels, such as grinding, crushing, milling, drying, dust extraction, and pressing, it seems necessary to pay special attention to safety in conducting such activities (Elsheikh *et al.*, 2022; Haque *et al.*, 2019; Hilary *et al.*, 2021; Yang *et al.*, 2015). An often overlooked aspect is the danger of dust explosiveness. It is important to become familiar with the basic mechanisms of dust explosions and methods of prevention and mitigation. The risk of dust explosions applies to a wide variety of industries that produce certain goods: wood and paper products (dust generated during, for example, sawing and cutting), foodstuffs and grains (*e.g.*, flour), metal products (*e.g.* metal dusts), power generation (*e.g.*, coal dust, wood), chemical sector (*e.g.*, pharmaceuticals and pesticides), mining (*e.g.*, coal, sulfur), and textile manufacturing (*e.g.*, cotton) (Amyotte and Eckhoff, 2010; Wei *et al.*, 2020). Despite a great deal of research and the introduction of specific preventive measures, technological developments are influencing the emergence of new risks that require constant analysis. Modification of technological processes in the production of certain materials often involves a change in their physical properties. If these changes affect the particle size and the level of moisture in the dust, the explosion risk of such materials may change (Castells *et al.*, 2021a, b). It should be noted that dust explosions are initiated by the rapid and sudden ignition of dust that rises in the air, along with a large increase in pressure and the presence of a shock wave. A dust explosion is possible if in a certain space there is a mixture of air with small dust particles, the concentration of which reaches a certain threshold in the presence of an ignition source. Dust explosions are very serious threats to human life and health, which is why it is so important to follow certain safety procedures along with modifying and updating them in the context of technological developments (Amyotte and Eckhoff, 2010; Abbasi and Abbasi, 2007; Eckhoff, 2009; Yuan *et al.*, 2023).

Using the pyrolysis process to manage agricultural residues such as straw has many benefits that can positively impact the energy industry economy and the environment. Such action can reduce the consumption of fossil raw materials, create new biofuel alternatives, or reduce the amount of waste in landfills. Limited resources of non-renewable

energy sources and intensification of energy consumption are among the most significant threats in modern society. The search for renewable materials for fuel production and the development of new technologies based on a sustainable development strategy can be a solution to the current crises of energy production and energy security. Recent years have seen an intensification of global research dedicated to the feasibility of disposal of waste generated in the industrial and agricultural sectors. Agricultural waste, due to its high yield and renewability, can be an attractive material for green fuel production. At the same time, it should be noted that a sizable portion of waste from this sector is not used but incinerated or left in fields. It is worth mentioning here that these materials have great potential for the production of solid biofuels with higher calorific value, such as biochars and torrefied biomasses. The use of various types of thermochemical transformations involves appropriate handling and safety procedures. The purpose of the research was to identify the basic parameters of dust explosion risk in the context of the use of crop straw as energy and biochar materials, along with an assessment of the impact of the pyrolysis process.

## 2. MATERIALS AND METHODS

### 2.1. Research object

To produce biochar, wheat, oat, and triticale straw were used separately. The research material was obtained from the owner of a farm located in Strzyżów District (Podkarpackie Province). The material intended for testing was dried to a moisture level of less than 10% and then crushed to a fraction of less than 10 mm.

### 2.2. Pyrolysis process

The pyrolysis process was conducted using a retort furnace FCF 2R designed for heat treatment in the atmosphere of inert gas, equipped with a post-process gas cooler with a water well (CZYŁOK, Jastrzębie-Zdrój, Poland).

Pyrolysis tests of the pellet families were carried out at temperatures of 400, 450, and 500°C respectively for 5, 10 and 15 min in a nitrogen atmosphere of 99.99% purity with a gas flow of 10 l min<sup>-1</sup>. The obtained pyrolysates were then sifted through a sieve with a diameter of holes equal to 1 mm. In order to remove potential contaminants, the samples were rinsed several times with distilled water and then dried for 12 h (at 80°C).

### 2.3. Analysis of samples

Basic physical and chemical parameters of the analyzed materials, *e.g.* the total content of carbon, ash, nitrogen, hydrogen, and volatile substances and the calorific value were determined. Analyses of the contents of ash and volatile substances in the samples were performed using a thermogravimetric method, with a TGA 701 apparatus from LECO (LECO Corporation, Saint Joseph, MI,

U.S.A.). The contents of total carbon, hydrogen, and nitrogen were determined using a TrueSpec CHN analyzer from LECO (LECO Corporation, Saint Joseph, MI, U.S.A.). In order to determine the calorific value of the materials analyzed, the AC500 calorimeter from LECO was used.

The analyses of dust explosiveness were carried out using a KSEP20 device equipped with the control unit KSEP 310 (Kuhner AG, Basel, Switzerland). The device has a test chamber in the form of a ball with a volume of 20 dm<sup>3</sup>. Dissipation of explosion heat and provision of thermostatically controlled test temperatures is provided by the water jacket

The analyzed dust is dispersed under pressure with use of an inlet valve, which is opened and closed pneumatically. The ignition source is provided by two chemical igniters with energy of 5kJ each, located in the central part of the sphere. The course of the process parameters is recorded with use of Kistler pressure piezoelectric sensors. As the result of the analyses, the maximum explosion pressure  $P_{max}$  was determined as the highest recorded explosion pressure of the combustible mixture in the form of a mixture of combustible material with air. This parameter, along with the value of the maximum increase in pressure over time  $(dp/dt)_{max}$ , is used to determine the  $K_{st,max}$  explosiveness class. This parameter is a determinant of European standards, which define the division of combustible dust according to EN14034 (Stelte *et al.*, 2011). The parameter was estimated from the following equation:

$$K_{max} = K_{st} = \sqrt[3]{V \left( \frac{dp}{dt} \right)_{max}} = 0.271 \left( \frac{dp}{dt} \right)_{max}$$

$K_{st,max}$  – explosivity index (MPa s<sup>-1</sup>),  $V$  – volume of the test chamber,  $(dp/dt)_{max}$  – indicator of maximum explosion pressure gain.

The value of the explosive index is classified according to the values shown in Table 1, where the St1 class indicates a material that is not very susceptible to explosion,

**Table 1.** Dust explosion classes (EN14034, Part 2)

Explosion class	$K_{st,max}$ value (MPa s <sup>-1</sup> )
St1	≤ 200
St2	200-300
St3	> 300

the St2 class indicates moderate susceptibility to explosion, while the St3 class indicates high susceptibility to explosion hazard.

Samples of biomass and biochars were subjected to laboratory analyses using current analytical standards (Table 2).

#### 2.4. Names of tests

For further identification, biomass samples were described using symbols depending on the type of material, temperature, and duration of the pyrolysis process: W – wheatstraw, O – oat straw, T – triticale straw, 1 – pyrolysis (temp. 400°C, 5 min); 2 – pyrolysis (temp. 400°C, 10 min); 3 – pyrolysis (temp. 400°C, 15 min); 4 – pyrolysis (temp. 450°C, 5 min); 5 – pyrolysis (temp. 450°C, 10 min); 6 – pyrolysis (temp. 450°C, 15 min); 7 – pyrolysis (temp. 500°C, 5 min); 8 – pyrolysis (temp. 500°C, 10 min); 9 – pyrolysis (temp. 500°C, 15 min).

For instance, W – non-heat-treated wheat straw, T6 – triticale straw with pyrolysis at 450°C and a time of 15 min.

#### 2.5. Statistical analysis

The effects of experimental factors reflected by the relevant parameters and the relationships between them were examined by Analysis of Variance (ANOVA) with use of Duncan's test. In order to compute the statistical analyses, STATISTICA version 12.0 (StatSoft Inc., Tulsa, OK, USA) was applied. A significance threshold of ≤ 0.05 was set for all analyses. The data were analyzed separately for each type of materials.

**Table 2.** Parameters analyzed according to the Polish Standards PN

Parameter	Research method	
Content of carbon, nitrogen and hydrogen	PN-EN ISO 16948:2015-07	Solid biofuels – Determination of total carbon, hydrogen and nitrogen content
Ash content	PN-EN ISO 18122:2023-05	Solid biofuels – Determination of ash content
Calorific value	PN-EN ISO 18125:2017-07	Solid biofuels – Determination of calorific value
Maximum explosion pressure $P_{max}$	PN-EN 14034-1+A1:2011	Determination of explosion characteristics of dust clouds – Part 1: Determination of the maximum explosion pressure $p_{max}$ of a dust cloud
Maximum rate of pressure $(dp/dt)_{max}$	PN-EN 14034-2+A1:2011	Determination of explosion characteristics of dust clouds – Part 2: Determination of the maximum rate of increase of explosion pressure $(dp/dt)_{max}$ of a dust cloud

### 3. RESULTS AND DISCUSSION

Currently, due to the search for energy alternatives, agricultural waste biomass, among others, is being used as a substrate for the pyrolysis process. Most often it is surplus straw from different varieties of grain, such as wheat or oats. Table 3 summarizes the percentages of total nitrogen, total carbon, hydrogen, ash, and volatile matter in untreated biomass and in biochar produced from wheat straw. For the control sample, the percentage of total nitrogen was 0.22%. In turn, the proportion of this element in the pyrolysate ranged from 0.95% in the biochar produced within 5 min at 400°C to 0.49% in the case of higher parameters. The total carbon content in the pyrolysate ranged from 68.32 to 70.77%, while that in the control sample was lower at 46.01%. The highest hydrogen content (6.11%) was recorded in the raw biomass, while the content of this element in the biochar ranged from 3.25 to 4.26%. The ash content in the wheat straw and the biochars produced from the material ranged from 3.13 to 16.5%. The lowest averaged ash content was recorded in the control sample, while the highest level was exhibited by biochar produced at 500°C for 15 min. There were statistically significant differences in the ash content in the biochar between the untreated biomass and the pyrolysates.

Table 4 shows the analytical results for oat straw and the pyrolysates produced from this type of biomass. The nitrogen content ranged from 0.38 in the control sample to 1.05% in the biochar produced by pyrolysis within 5 min at 400°C. In the biochar produced from oat straw, the total carbon had the highest percentage that ranged from 62.68 to

66.8%. The lowest percentage content of this element was recorded in the thermally unprocessed oat straw. In this biomass, the hydrogen content was 6.2%, while in the biochar it ranged from 3.48 to 4.41%. The thermally unprocessed oat straw had 4.35% ash content. Among the pyrolysates prepared from this type of straw, the lowest ash content (6.2%) was found in the biochar prepared within 5 min at 400°C. The highest percentage of ash content (17.52%) was determined in the pyrolysate produced within 15 min at 500°C.

Table 5 summarizes data on pyrolyzed triticale straw. In the case of the thermally unprocessed triticale straw, the carbon content was 44.82%. On the other hand, among the pyrolysates, the content of this element oscillated between 64.09% in the biochar produced within 5 min at 400°C and 70.33% in the material prepared within 15 min at 500°C. The results of the control sample were statistically significantly different from those of the biochar. The highest hydrogen content (6.17%) was again exhibited by straw that was not subjected to pyrolysis. The biochar from the triticale straw had lower content of this element; for 500°C and the individual times, the values were 3.57, 3.47, and 3.38%. The analyses of the percentage of nitrogen showed the highest percentage of this element (0.99%) in the biochar produced at 400°C for 5 min. The percentage of nitrogen in the non-pyrolyzed triticale straw was the lowest of all the samples analyzed, at 0.27%. The ash content in the resulting pyrolysates ranged from 5.86 to 17.01%. The lowest percentage was recorded in the control sample, while the highest value was recorded in the biochar produced for 15 min at 500°C.

**Table 3.** Content of selected elements and ash in wheat straw and biochars produced from it

Material	N	C	H	Ash
	(%)			
W	0.22 <sup>a</sup> ± 0.01	46.01 <sup>a</sup> ± 0.25	6.11 <sup>d</sup> ± 0.01	3.13 <sup>a</sup> ± 0.14
W1	0.95 <sup>c</sup> ± 0.05	64.37 <sup>b</sup> ± 0.25	4.26 <sup>c</sup> ± 0.02	5.53 <sup>b</sup> ± 0.07
W2	0.87 <sup>d</sup> ± 0.03	68.32 <sup>bc</sup> ± 0.09	4.22 <sup>c</sup> ± 0.02	6.63 <sup>b</sup> ± 0.14
W3	0.84 <sup>d</sup> ± 0.02	68.47 <sup>bc</sup> ± 0.10	4.23 <sup>c</sup> ± 0.02	8.30 <sup>c</sup> ± 0.12
W4	0.67 <sup>c</sup> ± 0.03	68.2 <sup>bc</sup> ± 0.16	3.85 <sup>b</sup> ± 0.02	9.48 <sup>c</sup> ± 0.13
W5	0.62 <sup>c</sup> ± 0.01	68.72 <sup>bc</sup> ± 0.09	3.84 <sup>b</sup> ± 0.01	11.60 <sup>d</sup> ± 0.14
W6	0.63 <sup>c</sup> ± 0.01	69.24 <sup>bc</sup> ± 0.11	3.84 <sup>b</sup> ± 0.02	12.76 <sup>de</sup> ± 0.13
W7	0.1 <sup>b</sup> ± 0.02	69.66 <sup>bc</sup> ± 0.16	3.41 <sup>a</sup> ± 0.02	13.92 <sup>e</sup> ± 0.19
W8	0.5 <sup>b</sup> ± 0.02	70.28 <sup>bc</sup> ± 0.10	3.40 <sup>a</sup> ± 0.02	15.45 <sup>f</sup> ± 0.10
W9	0.49 <sup>b</sup> ± 0.03	70.77 <sup>c</sup> ± 0.11	3.25 <sup>a</sup> ± 0.01	16.50 <sup>f</sup> ± 0.07

Statistically significant differences marked by different letters ( $p \leq 0.05$ ). Differences between average values marked with the same letters are not statistically significant at the level of  $p \leq 0.05$  according to the Duncan test. The data were analyzed separately for each type of materials.

**Table 4.** Content of selected elements and ash in oat straw and biochars produced from it

Material	N	C	H	Ash
	(%)			
O	0.38 <sup>a</sup> ± 0.01	42.31 <sup>a</sup> ± 0.10	6.2 <sup>d</sup> ± 0.01	4.35 <sup>a</sup> ± 0.04
O1	1.05 <sup>c</sup> ± 0.05	62.68 <sup>b</sup> ± 0.20	4.41 <sup>c</sup> ± 0.01	6.20 <sup>b</sup> ± 0.07
O2	0.98 <sup>d</sup> ± 0.03	64.67 <sup>bc</sup> ± 0.08	4.34 <sup>c</sup> ± 0.01	7.47 <sup>b</sup> ± 0.07
O3	0.94 <sup>d</sup> ± 0.02	65.15 <sup>bc</sup> ± 0.04	4.34 <sup>c</sup> ± 0.02	8.89 <sup>c</sup> ± 0.07
O4	0.77 <sup>c</sup> ± 0.03	66 <sup>bc</sup> ± 0.12	4.01 <sup>b</sup> ± 0.01	9.72 <sup>c</sup> ± 0.06
O5	0.73 <sup>c</sup> ± 0.01	66.67 <sup>bc</sup> ± 0.07	3.93 <sup>b</sup> ± 0.05	12.02 <sup>d</sup> ± 0.17
O6	0.73 <sup>c</sup> ± 0.01	67.41 <sup>bc</sup> ± 0.06	3.93 <sup>b</sup> ± 0.01	13.56 <sup>ef</sup> ± 0.09
O7	0.62 <sup>b</sup> ± 0.02	68.24 <sup>bc</sup> ± 0.10	3.73 <sup>a</sup> ± 0.18	14.85 <sup>f</sup> ± 0.07
O8	0.61 <sup>b</sup> ± 0.02	68.8 <sup>c</sup> ± 0.07	3.57 <sup>a</sup> ± 0.06	16.12 <sup>g</sup> ± 0.04
O9	0.59 <sup>b</sup> ± 0.03	68.78 <sup>c</sup> ± 0.18	3.48 <sup>a</sup> ± 0.01	17.52 <sup>h</sup> ± 0.04

Explanations as in Table 3.

**Table 5.** Content of selected elements and ash in triticale straw and biochars produced from it

Material	N	C	H	Ash
	(%)			
T	0.27 <sup>a</sup> ± 0.01	44.82 <sup>a</sup> ± 0.07	6.17 <sup>d</sup> ± 0.01	3.85 <sup>a</sup> ± 0.09
T1	0.99 <sup>c</sup> ± 0.01	64.09 <sup>b</sup> ± 0.06	4.35 <sup>c</sup> ± 0.01	5.86 <sup>b</sup> ± 0.07
T2	0.91 <sup>dc</sup> ± 0.03	67.05 <sup>bc</sup> ± 0.06	4.4 <sup>c</sup> ± 0.01	7.05 <sup>c</sup> ± 0.10
T3	0.87 <sup>d</sup> ± 0.02	67.37 <sup>bc</sup> ± 0.06	4.3 <sup>c</sup> ± 0.02	8.60 <sup>d</sup> ± 0.09
T4	0.7 <sup>c</sup> ± 0.03	67.66 <sup>bc</sup> ± 0.14	3.95 <sup>b</sup> ± 0.02	9.60 <sup>d</sup> ± 0.05
T5	0.66 <sup>c</sup> ± 0.01	68.25 <sup>bc</sup> ± 0.05	3.91 <sup>b</sup> ± 0.03	11.81 <sup>e</sup> ± 0.15
T6	0.66 <sup>c</sup> ± 0.01	68.98 <sup>bc</sup> ± 0.04	3.9 <sup>b</sup> ± 0.01	13.16 <sup>f</sup> ± 0.09
T7	0.55 <sup>b</sup> ± 0.02	69.51 <sup>bc</sup> ± 0.09	3.57 <sup>a</sup> ± 0.12	14.39 <sup>f</sup> ± 0.06
T8	0.54 <sup>b</sup> ± 0.02	70.1 <sup>c</sup> ± 0.08	3.47 <sup>a</sup> ± 0.06	15.78 <sup>g</sup> ± 0.06
T9	0.52 <sup>b</sup> ± 0.03	70.33 <sup>c</sup> ± 0.05	3.38 <sup>a</sup> ± 0.01	17.01 <sup>g</sup> ± 0.05

Explanations as in Table 3.

Statistically significant differences were found between the raw biomass and the pyrolysates produced from triticale straw.

The pyrolysis process had a statistically significant effect on the content of the elements in biochars in relation to untreated biomass. The average carbon content in all the pyrolysates was 67%. In their study, Enders *et al.* (2012) noted that as the temperature of the pyrolysis process increased, the amount of total carbon in the final product also increased. In addition, by analyzing biochar produced from beech wood, they determined the carbon content at the level of 50% (Enders *et al.*, 2012). As shown by Saletnik

*et al.* (2016), the carbon content in rapeseed straw biochar produced at 400°C for 10 min was 59.01% (Saletnik *et al.*, 2016). Also Zheng *et al.* (2011) proved that, regardless of the substrate used in the pyrolysis process, the total carbon content increased along with the increase in the temperature of the reaction (Zheng *et al.*, 2011). On the other hand, Aqsha *et al.* (2017) found in their study that the amount of hydrogen and nitrogen decreases with increasing values of pyrolysis parameters regardless of the substrate subjected to the pyrolysis process, which is confirmed by the present results (Aqsha *et al.*, 2017). As reported by Zheng *et al.* (2011), the hydrogen content in biochar produced from

corn kernels decreased by about 25% when the pyrolysis process operating temperature was changed from 400 to 500°C (Zheng *et al.*, 2011). Comparing the results obtained to Aqsha *et al.* (2017), the ash content in the thermally non-converted biomass was comparable at 4.69% (Aqsha *et al.*, 2017). In her work, Malińska (2012) reports that the ash content in biochar produced from such plants as rapeseed and sunflower at 500°C is in the range of 21-28.9% (Malińska, 2012). A higher amount of ash in this type of straw was shown by Grubor *et al.* (2016), where the content of this compound was 6.07% (Grubor *et al.*, 2016).

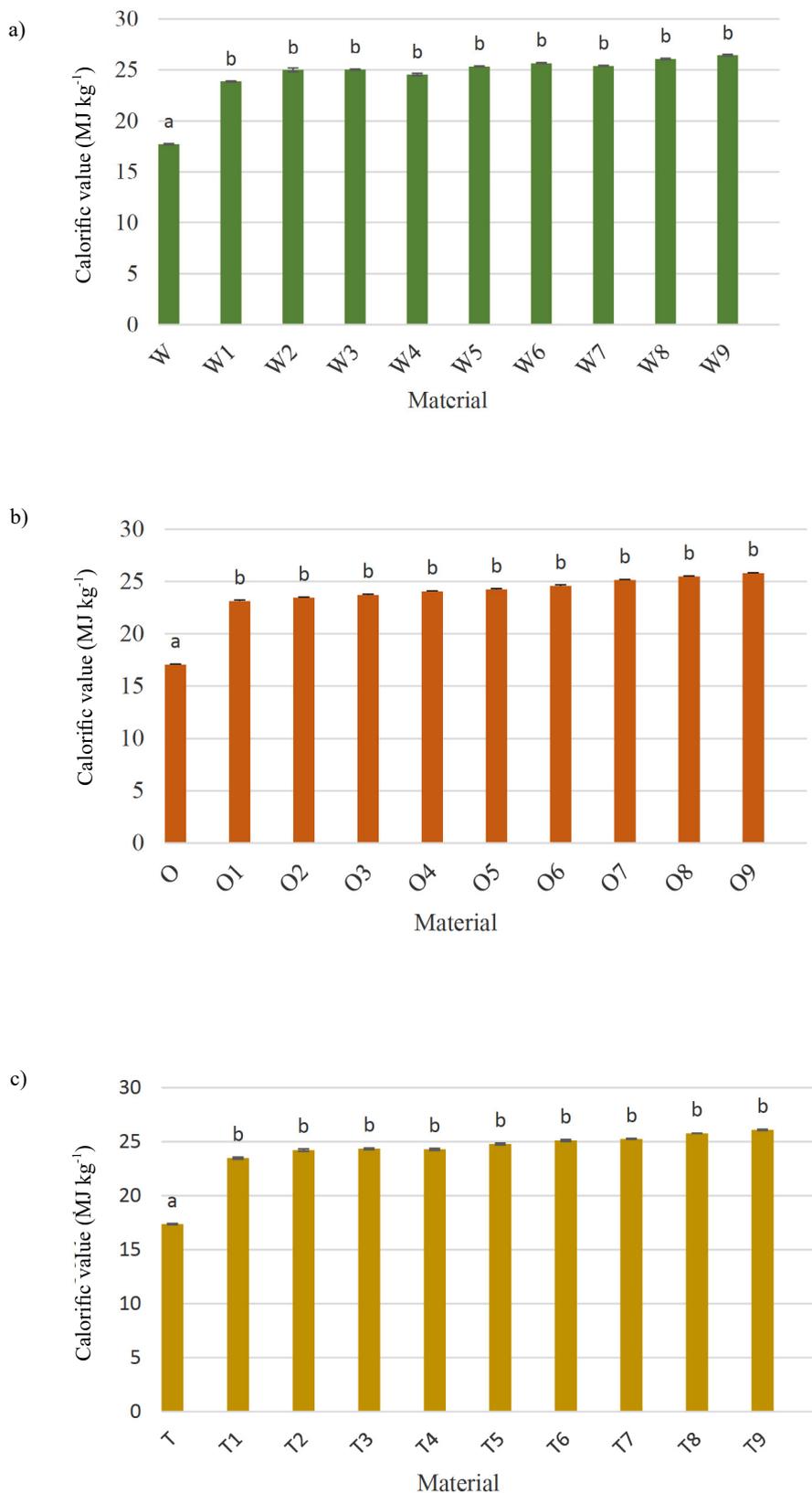
Figure 1 presents the results of the calorific value of the biomass and the biochar obtained from the wheat straw (a), oat straw (b), and triticale straw (c). The calorific value of the wheat, oat, and triticale straw pyrolysates was 25.26, 24.4, and 24.8 MJ kg<sup>-1</sup>, respectively. In contrast, the results in the non-heat-converted straw ranged from 17.07 MJ kg<sup>-1</sup> in the oat straw to 17.71 MJ kg<sup>-1</sup> in the sample containing wheat straw.

The results of the calorific value of biochars were statistically significantly different from those of the control samples. The highest calorific value for all analyzed materials was obtained after applying the pyrolysis process at 500°C for 15 min. Based on the calorific value obtained in biochars produced from the wheat straw, oat straw, and triticale straw, the optimal conditions for the pyrolysis process can be selected. Given the statistically insignificant differences in the presented results between the different pyrolysates, it should be assumed that the most favorable conditions would be values of temperature and time at 400°C and 5 min, respectively. The listed parameters were achieved in the shortest time and with the least use of energy, which determines their use as optimal values. Analyzing the presented data, it can be concluded that the parameters for conducting the pyrolysis process for the tested types of straw do not have a significant impact on the calorific value of biochar that was the final product. Also, the type of substrate used in this process (three types of straw) did not affect the energy characteristics, as confirmed in their study by Grubor *et al.* (2016). As reported by Malińska (2012), the calorific value of hard coal and lignite ranged from 21 to 25 MJ kg<sup>-1</sup> (Malińska, 2012). The results presented in this paper showed that the similar calorific value of biochars produced by pyrolysis of waste agricultural biomass in relation to the most commonly used fossil fuels justifies the use of biochar as an alternative source for electricity and heat production. A slight difference in results was presented by Mirowski *et al.* (2018), where unprocessed wheat straw showed a calorific value of 17.3 MJ kg<sup>-1</sup> (Mirowski *et al.*, 2018). In contrast, Portarapillo *et al.* (2021) report a calorific value for wheat straw at the level of 17.9 MJ kg<sup>-1</sup> (Portarapillo *et al.*, 2021). The literature reports that the calorific value of biochar made from wheat straw ranges from 20.3 to 21.4 MJ kg<sup>-1</sup> (Mohanty *et al.*, 2013). Malińska (2012) examined the calorific value of biochar made from

palm oil residues and recorded the calorific value of this material at 17 MJ kg<sup>-1</sup> (Malińska, 2012). As shown in our previous study, a higher calorific value (27.7 MJ kg<sup>-1</sup>) was exhibited by biochars produced from material with higher lignin content, such as cherry wood (Saletnik *et al.*, 2019). In a study on the torrefaction of plant biomass, Bajcar *et al.* (2020) determined the calorific value of wheat straw at 17.51 MJ kg<sup>-1</sup> (Bajcar *et al.*, 2020). Anaerobic thermal processing at 300°C for 60 minutes allowed this value to increase to 21.46 MJ kg<sup>-1</sup>. In contrast, Park *et al.* (2014) studied the slow pyrolysis of rice straw and reported that an increase in the temperature of the process from 300 to 700°C resulted in a decrease in the calorific value of the material from 16.6 to 13.6 MJ kg<sup>-1</sup> (Park *et al.*, 2014). On the other hand, in a study conducted by Sedmihradská *et al.* (2020), biochar from barley straw prepared at 500°C was characterized by a calorific value at the level of 25.5 MJ kg<sup>-1</sup> (Sedmihradská *et al.*, 2020).

The results of the analysis of the maximum pressure showed similar dependencies of the changes in this parameter after applying the pyrolysis process for all the tested materials. The P<sub>max</sub> value of dust of thermally untreated materials was in the range from 7.07 to 7.44 MPa. The pyrolyzed materials were characterized by higher P<sub>max</sub> values, with the highest value at 9.03 MPa recorded for the wheat straw pyrolysate dust (for pyrolysis parameters such as 500°C and time of 10 min). It was noted that both the temperature and the time of the pyrolysis process induce changes in the maximum explosion pressure of the obtained biochar materials. Similar changes in the tested materials were also determined for the indicator of maximum pressure rise rate. The average value of this parameter for dust from all types of thermally untreated straw tested was 273.05 MPa s<sup>-1</sup>. Subjecting the three types of straw to pyrolysis induced an average increase in the value of the (dp/dt)<sub>max</sub> ratio to a maximum of 333.21 MPa s<sup>-1</sup>. As before, the highest values were obtained in the variant using the pyrolysis process with a temperature of 500°C and a time of 15 min. Pólka and Kukfisz (2015) report that the maximum pressure rise rate parameter is considered to be the best indicator of dust explosion strength. In their paper on the explosive parameters of eucalyptus dust in the wood and furniture industry, the authors determined the value of (dp/dt)<sub>max</sub> to be 314.72 MPa s<sup>-1</sup>. In contrast, the maximum explosion pressure of eucalyptus dust was characterized by the value equal to 9.78 MPa (Pólka and Kukfisz, 2015). Kukfisz (2018) reports that a mixture of coal and biomass dust can be far more dangerous than individual dusts of these materials. The author reports that the value of P<sub>max</sub> for biomass-derived dusts and their mixtures ranges from 5.5 to over 8 MPa (Kukfisz, 2018) (Table 6).

The obtained values of dust explosiveness indices allowed classification of all the tested materials, *i.e.* wheat, oat, and triticale straw, and the pyrolysates produced from the biomasses as materials with low susceptibility to



**Fig. 1.** Calorific value of unprocessed biomass and biochars produced from wheat straw depending on the parameters of the pyrolysis process. Differences between average values marked with the same letters are not statistically significant at the level of  $p \leq 0.05$  according to the Duncan test. The data were analyzed separately for each type of materials.

**Table 6.** Maximum rate of pressure buildup and the maximum explosion pressure of dust from the straw tested and the pyrolysates prepared

Material	$P_{max}$	$(dp/dt)_{max}$
	(MPa)	(MPa s <sup>-1</sup> )
W	7.44	291.80
W1	8.17	302.43
W2	8.41	304.19
W3	8.65	307.01
W4	8.41	322.83
W5	8.73	345.27
W6	8.83	353.26
W7	8.67	330.23
W8	8.87	346.28
W9	9.03	356.09
O	7.30	270.39
O1	8.01	280.23
O2	8.25	281.86
O3	8.47	284.48
O4	8.25	299.13
O5	8.55	319.93
O6	8.65	327.33
O7	8.49	305.99
O8	8.69	320.87
O9	8.85	329.96
T	7.07	256.97
T1	7.77	266.33
T2	8.00	267.88
T3	8.22	270.37
T4	8.00	284.29
T5	8.29	304.06
T6	8.39	311.09
T7	8.24	290.81
T8	8.43	304.95
T9	8.58	313.59

explosion (St1 hazard class). The dust explosion indices for most of the pyrolysates obtained differed statistically significantly from those of the thermally unprocessed straw. Despite the increase in the value of the studied parameter with the change in pyrolysis process parameters, *i.e.* the increase in temperature and time, there were no statistical differences in this parameter between the different pyrolysates. Each time, the highest  $K_{st\ max}$  value was identified for biochar prepared at 500°C for 15 min. The average value of the dust explosion index for all the types of straw analyzed was 71.17 MPa s<sup>-1</sup>, while the average value for all the biochars obtained was 83.7 MPa s<sup>-1</sup>. Portarapillo *et al.* (2021) report

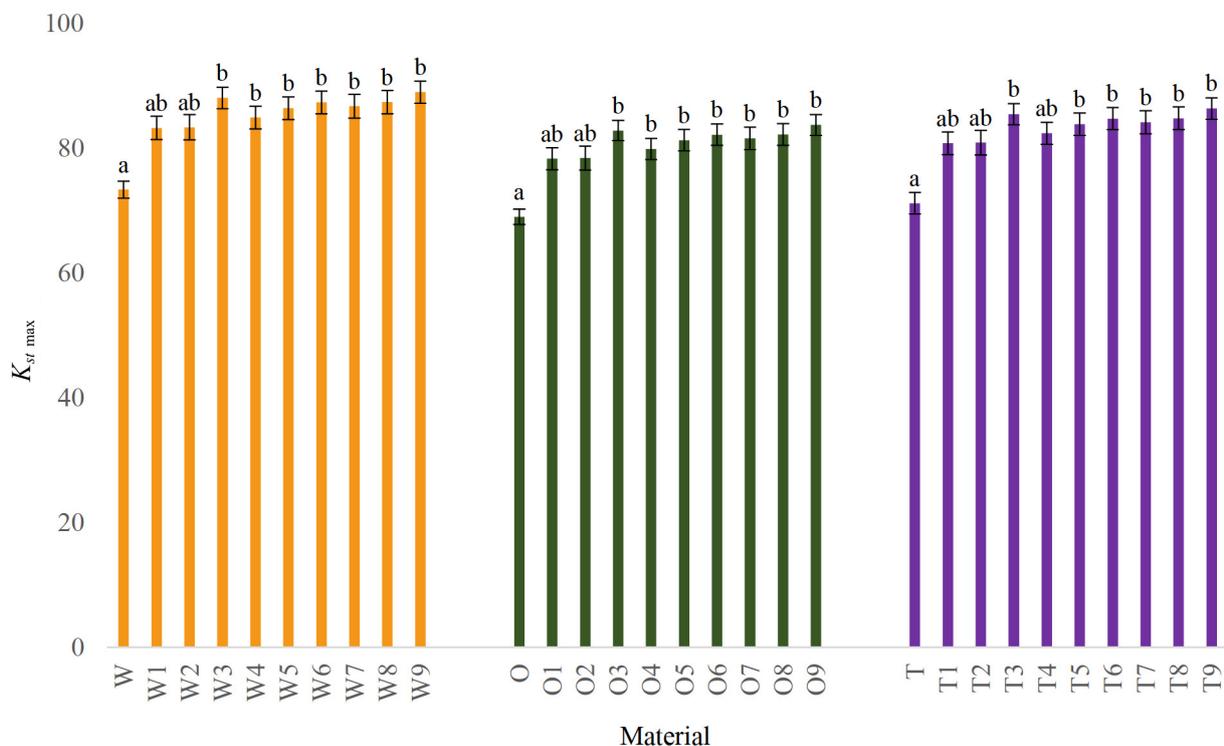
a wide range of  $K_{st\ max}$  values for dust from various types of plant biomass. In the presented comparison, the dust explosion rate was characterized by values of 23, 72, 98, and 194.4 MPa s<sup>-1</sup> for rapeseed straw, barley straw, walnut shells, and pine sawdust, respectively (Portarapillo *et al.*, 2021). In the paper by Saeed *et al.* (2016), the researchers determined the value of the explosive index for wheat straw to be 81.7 MPa s<sup>-1</sup>, which is slightly higher than in the study presented in this paper. The authors also report values for two coal samples, *i.e.* Colombian Coal and Kellingley Coal, where the  $K_{st\ max}$  values were quite varied and were 122.9 and 78.2 MPa s<sup>-1</sup>, respectively (Saeed *et al.*, 2016). In a study of different nano-sized coal dust, Tan *et al.* (2020) classified the tested material into St1 and St2 Explosivity Classes – low and medium levels of explosion hazard. The  $K_{st\ max}$  parameter for coal dust was determined to range from 30.7 to 223.5 MPa s<sup>-1</sup> (Tan *et al.*, 2020) (Fig. 2).

#### 4. CONCLUSIONS

The study found that wheat, oat, and triticale straw can be attractive materials for the production of high-energy biochar. By modifying the parameters of the pyrolysis process, changes in the calorific value of the biochar obtained were noted, while the differences in this parameter were not statistically significant. The highest values were obtained for the straw pyrolysis process at 500°C lasting for 15 min, and the average increase in the calorific value for all biochars from the three types of straw in relation to the thermally untreated biomass was 30%. It was found that the total carbon content increased along with the increasing time and temperature of the pyrolysis process, and a statistically significant increase in the ash content in the pyrolysates was noted.

The most important result of the study was to determine the dust explosion hazard of the obtained biochar materials. This parameter is often overlooked in the characteristics of new alternative biofuels, and the literature does not contain much information in this regard for pyrolysates derived from biomass from the agricultural sector. There was a positive correlation between the increase in the temperature and time of the anaerobic thermal treatment of biomass and the dust explosion rate. However, these changes did not affect the classification of the biochars, and all the materials obtained were classified as class one – not very prone to explosiveness. The highest recorded explosivity index value was exhibited by the wheat straw biochar (500°C, 15 min) and was at the level of 88.99 MPa s<sup>-1</sup>.

Considering the energy sector and its use of agricultural residues, straw can be divided into freshly harvested straw and gray straw, which seems to be a more attractive energy material. On the other hand, it is important to rationally manage this raw material taking into account its resources and surplus, which can be used for energy purposes. The studies conducted indicate that straw can be used to produce



**Fig. 2.** Dust explosive index of the tested straw and prepared pyrolysates. Explanations as on Fig. 1.

high-quality biochar, which can be used directly for energy purposes or as a feedstock for the production of other solid biofuels. It should also be emphasized that the use of the straw pyrolysis process does not increase the risk of dust explosion during straw processing and modification.

**Conflicts of Interest:** The Authors do not declare any conflict of interest.

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