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REVEALING THE INFLUENCE OF AGE AND FRACTIONAL COMPOSITION OF WOODY BIOMASS ON THE KINETIC CHARACTERISTICS OF THERMAL DECOMPOSITION

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This study investigates processes related to thermal decomposition and direct combustion of woody biomass derived from the fast-growing energy willow Salix fragilis. The task addressed is predetermined by the lack of a sufficient database on kinetic parameters required for the efficient utilization of woody biofuel in modern boiler systems, particularly under conditions of transitioning from fossil to renewable energy sources.

The thermal degradation pattern of woody biomass of different ages and particle-size distributions was explored in detail using differential thermogravimetry (DTG) and differential thermal analysis (DTA). The results demonstrate empirical dependences of relative mass loss of samples at heating, which made it possible to identify the characteristic stages of thermal decomposition and the intensity of mass transfer.

The clearly observed influence of biomass age and particle-size distribution enabled identification of the key kinetic features that directly affect the combustion rate and completeness of fuel conversion. These differences provide a more accurate prediction of fuel behavior in actual power units and lay the basis for forming the primary database of combustion kinetic constants. The results are attributed to differences in the structural organization of the wood, the biomass age, as well as the content of volatile components in samples of different age groups.

The practical application of the established dependences is relevant for the design and optimization of boilers operating on comminuted woody biomass. The defined parameters make it possible to optimize fuel particle-size composition, ensure a rational residence time of biomass particles in the combustion zone, and improve the energy efficiency of boiler units when replacing traditional fuels with renewable raw materials

Keywords: energy willow (Salix fragilis), thermal analysis, thermal decomposition, thermogram, derivatograph

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1. Introduction

Under current conditions for energy sector evolution, the need to find stable and environmentally friendly energy sources is increasing. The main factor determining the need for such research is the constant increase in prices for traditional energy resources, in particular, natural gas and oil, which currently remain key fuels in the global energy balance. In addition to economic factors, the environmental component is gaining significant importance: the implementation of EU legislation on reducing harmful emissions and complying with environmental standards requires a gradual transition to renewable energy sources [1]. This necessitates the development of a balanced energy sector that enables the rational use of resources, reduces the environmental burden, and contributes to increasing the country's energy independence.

A special role in modern energy policy belongs to bioenergy, which includes the generation of energy from biomass and organic waste. The acceleration of the introduction of biofuels is influenced by economic incentives, state support, and environmental requirements [2]. In the EU countries, more than 12.5% of thermal energy is provided by alternative fuels, of which about 77% is utilized through waste processing, and the rest is provided by biomass [3]. Biomass is recognized as one of the most environmentally neutral and renewable energy sources, providing about 14% of the world's primary energy consumption. Its global annual production reaches approximately 2 billion tons of equivalent fuel, which allows it to occupy the fourth place among all energy sources [4].

In world practice, biomass is mainly used by direct combustion to obtain thermal and electrical energy or biogas. The most economical approach is to use local resources with low cost, such as wood residues, straw, and sunflower husks [5]. In some EU countries, fossil fuels have almost completely been replaced by alternative sources at industrial facilities; the possibilities of extending such technologies to boiler houses and gas piston plants are now being studied [6].

International practice shows significant variability in the use of biomass: in developing countries, the share of biomass in energy consumption can exceed 30%, and in some cases reach 50–80% [7]. Gross domestic biomass consumption in 2024 and the forecast for 2050 for the 27 EU countries and the UK in tons of oil equivalent (toe) are shown in Fig. 1.

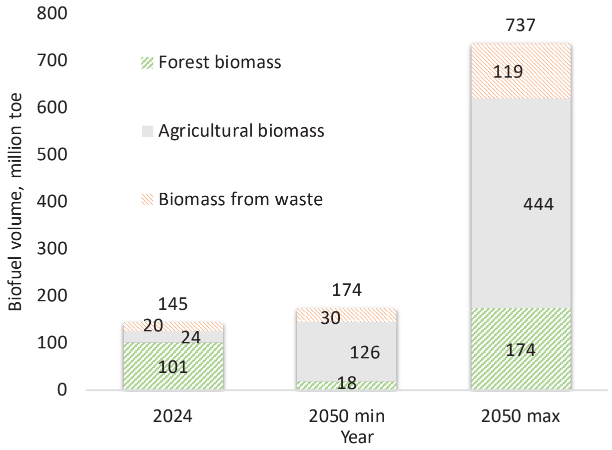

Fig. 1. Gross domestic energy consumption from biomass in 2024 and potential for 2050 for the European Union and the United Kingdom (in million toe). Based on data from [7, 8]

In the EU countries [8], the share of biomass among primary energy resources is as follows: Finland – 23%, Sweden – 18%, Austria – 12%, Denmark – 8%, Germany – 6%. Given the agricultural potential and waste volumes, all these countries have favorable conditions for the development of bioenergy.

Biomass is characterized by its flexibility of application, meeting the needs of heating, cooling, electricity generation, and transport. In the EU, about 85% of thermal energy from renewable sources is produced from biomass [9]. Despite the dominance of wood, an increase in the share of agricultural biomass and organic waste is predicted, which would make it possible to scale the bioenergy sector without a negative impact on forest resources.

Analysis of wood processing technologies reveals that thermal methods are the most appropriate: combustion, gasification, and pyrolysis, with combustion indicators exceeding the characteristics of other methods.

Thus, the growth of the share of biomass in the energy balance necessitates an in-depth study of the processes of its thermal destruction. The efficiency of burning wood biomass significantly depends on the age of plant raw materials and its fractional composition; the limited availability of reliable kinetic data makes it difficult to predict the progress of heat and mass transfer processes in boiler plants.

Therefore, it is a relevant task to carry out studies aimed at establishing kinetic characteristics of the thermal decomposition of wood biomass.

2. Literature review and problem statement

The modern energy sector is under significant pressure due to the need to reduce the use of fossil resources and transition to sustainable, renewable energy sources. One of the most accessible and environmentally sound types of biofuels in the world is woody biomass, in particular fast-growing energy crops, such as energy willow (*Salix fragilis*). The increased interest in their use is due to both the high growth rate of biomass and the ability to provide stable volumes of fuel with minimal cultivation costs. Under these conditions, a thorough study on the processes of thermal conversion of biomass is relevant, which makes it possible to predict its behavior in energy units of various types.

Work [10] reports the results from thermogravimetric analysis of energy crops, which show that the process of thermal destruction of biomass has a multi-stage nature and significantly depends on the heating rate. It is shown that kinetic parameters can vary significantly depending on the type of plant raw material. However, in the cited work, issues related to the influence of biomass age and its fractional composition on the course of the thermal destruction process remained unresolved. A likely reason is difficulties associated with the high variability of the physicochemical properties of wood raw materials, as well as the limitations of laboratory methods for modeling real combustion conditions.

In [11], the thermal decomposition of wood was investigated with a division into the main structural components (hemicellulose, cellulose, lignin), which allowed for a deeper analysis of the decomposition mechanisms. However, the authors considered only one type of wood without taking into account age characteristics and the grain size composition of the fuel.

The results of the studies reported in [12] indicate that alternative fuels differ in physicochemical properties and combustion kinetics compared to fossil fuels, in particular particle size and moisture content. Grinding of alternative fuels is carried out less intensively to reduce energy costs, while coal has a fine-grained structure (~5% residues on a 90 μm sieve).

The studies described in [13] show that the combination of thermogravimetric analysis with gas analysis methods (TG-FTIR) makes it possible to link the kinetics of mass loss with the chemical composition of volatile products. However, such approaches are technologically complex and expensive, which limits their application for a wide range of bioenergy research.

Under these conditions, there is a need for a thorough study on the processes of thermal conversion of biomass, which could make it possible to understand its behavior in energy units, in particular in boilers of various designs. Despite the spread of biofuel combustion technologies, the kinetic parameters and mechanisms of thermal decomposition of wood of different ages and fractional composition have not yet been studied in detail. The lack of clear dependences complicates the optimization of combustion modes, leads to a decrease in energy efficiency, and deterioration of environmental performance of energy equipment.

Studies from a number of research institutions [14] have confirmed the technical feasibility and economic efficiency of recycling wood waste, including contaminated forest areas, by gasification and pyrolysis, to obtain environmentally friendly gaseous fuel. The most valuable waste is from stem wood, bark, wood rot, tree crowns, and straw, as evidenced by the results of studies in [15].

Efficiency and environmental safety are determined by the combustion parameters, in particular the kinetics of oxidation processes. The variability of the physicochemical properties of wood biomass, such as moisture, density, and ash content, makes it difficult to determine the optimal heat-technological mode of operation of boilers.

An option to overcome these difficulties involves the process of direct combustion of biomass (sunflower husk, wood particles) at industrial rotary kilns to replace natural gas in order to save energy resources. This is the approach used in [16]; however, biomass combustion should ensure a high degree of energy utilization through complete particle burnout.

All this gives grounds to argue that it is advisable to conduct a study aimed at determining the kinetic characteristics of combustion for further design of target fuels adapted for specific types of biofuels.

In [17], a comparative analysis of kinetic models of thermal destruction of different types of biomass is given, which confirms the significant influence of the type of raw material on the mechanism and rate of decomposition. However, the generalized nature of the study does not make it possible to directly use the obtained parameters for specific types of woody biomass, in particular energy willow.

The kinetic characteristics of combustion, such as the rate of heat release and temperature profile, significantly depend on the properties of the fuel. For the rational use of wood biomass, a comprehensive analysis of its energy characteristics is necessary, determination of thermal decomposition parameters and construction of mathematical models for predicting fuel behavior in real combustion conditions. Work [18] shows the possibility of using thermogravimetric data to estimate the kinetic parameters of biomass; however, the study mainly concerns agricultural waste and does not cover wood raw materials.

It is advisable to study the combustion reactions of wood biomass using methods of chemical thermodynamics and kinetics. To derive reliable kinetic dependences, information is required not only about the initial and final states but also about the reaction pathway, which is usually uncertain. Establishing a kinetic model and parameters makes it possible to calculate the reaction rate and determine the optimal conditions for its implementation in specific heat engineering systems.

There are a large number of methods for determining kinetic parameters, which are usually divided into three groups: differential, integral, and methods with variable heating rates. Integral approaches involve approximation of the integral form of the reaction rate equation (Reich, Kots-Redfern methods, and others) [19], but require at least two experiments with one sample, have an uncertain reaction order and high sensitivity to temperature fluctuations.

Differential methods are based on the use of the differential form of the equation describing the kinetics of thermal destruction, in particular the methods of Van't Hoff, Freeman-Carroll, Doyle, Jaco, and others [19]. The most universal and widespread is the Freeman-Carroll method, which is characterized by its simplicity of implementation, the possibility of determining kinetic parameters from a single thermogram, and its suitability for complex multi-stage thermal decomposition processes.

The results of research in this area can have a significant practical effect. They are able to provide scientifically based recommendations for the correct selection of fuel parameters, determination of the optimal duration of its stay in the combustion zone, as well as improvement of design solutions of boiler plants focused on the use of biomass. In addition, the establishment of kinetic characteristics makes it possible to increase the accuracy of mathematical models of thermal processes, which is important for the development of modern automatic combustion control systems.

Summarizing the above arguments, it can be stated that the construction of a reliable base of kinetic parameters for the biomass of fast-growing energy willow is an important element of the transition to an environmentally safe and energy-efficient economy.

Thus, our review of the literature [10–18] reveals a large body of research into the thermal conversion of biomass; however, the issue of the influence of the age and fractional composition of wood biomass on the kinetic characteristics of its thermal decomposition remains insufficiently studied. All this gives grounds to argue that it is advisable to conduct a study aimed at determining the kinetic characteristics of thermal conversion of the biomass of fast-growing energy willow in order to further use results for designing and optimizing boiler plants focused on renewable fuels.

3. The aim and objectives of the study

The aim of our study is to identify the influence of the age and fractional composition of wood biomass on the features of the thermochemical process of its conversion using modern methods, in particular differential thermogravimetry (DTG). This will make it possible to compile a reliable database of kinetic parameters for the thermal conversion of the fast-growing energy willow's biomass. This database is necessary for a well-founded choice of fuel parameters and determination of the optimal duration of its particles in the combustion zone. It will also contribute to the improvement of structural and regime solutions for boiler plants focused on the use of biomass. In addition, results could enable an increase in the accuracy of mathematical models of thermal processes for designing modern automatic combustion control systems.

To achieve the aim of the study, the following tasks were defined:

– to analyze the characteristics of the elemental and fractional composition of experimental samples of wood biomass;

– to investigate the features of thermal decomposition of crushed biomass of energy willow (*Salix fragilis*) of different ages and fractional composition using differential thermogravimetry (DTG) and differential thermal analysis (DTA) methods.

4. The study materials and methods

The object of our study is the processes of thermal decomposition and direct combustion of woody biomass from the fast-growing energy willow *Salix fragilis*.

The principal hypothesis assumes that the age characteristics and fractional composition of woody biomass *Salix fragilis* significantly affect the kinetic parameters of thermal destruction and combustion, and taking into account these factors makes it possible to increase the completeness of combustion and energy efficiency of heat engineering plants.

Before starting the study, it was assumed that the biomass samples are representative of the corresponding age groups of willow, their elemental and fractional composition is homogeneous within each series, and the processes of thermal destruction recorded under laboratory conditions adequately reflect the main regularities of real combustion.

In the process of research, simplifications were adopted related to the consideration of the thermal decomposition process under conditions of uniform heating, neglecting the influence of turbulence and mass transfer with the environment, as well as the reduction of complex multi-stage reactions to generalized kinetic dependences.

In the experimental part of the work, standardized approaches were used to determine the kinetic parameters of the thermal conversion of wood biomass. The studies were carried out using modern control and measuring equipment and laboratory facilities. The processing of the experimental data was carried out using Mathcad software (USA).

For a detailed experimental study on the thermal decomposition of crushed woody biomass, a comprehensive approach was used, combining the methods of differential thermogravimetry (DTG) and differential thermal analysis (DTA). The methodology is based on the gradual heating of biomass samples placed in a specialized electric furnace, with simultaneous registration of changes in the mass of the sample *dm* (thermogravimetric curve TG), the rate of mass change *dm*/*dt* (DTG curve), as well as the temperature dependence of these changes. Additional information about energy transformations in biomass during heating or cooling is provided by the DTA curve, which records the temperature difference between the sample under study and an inert standard using a differential thermocouple. This makes it possible to determine the enthalpy effects of reactions, including exo- and endothermic processes, which differ in the direction of the peaks on the DTA curve. To illustrate the methodology, an example of a derivatogram is shown in Fig. 2.

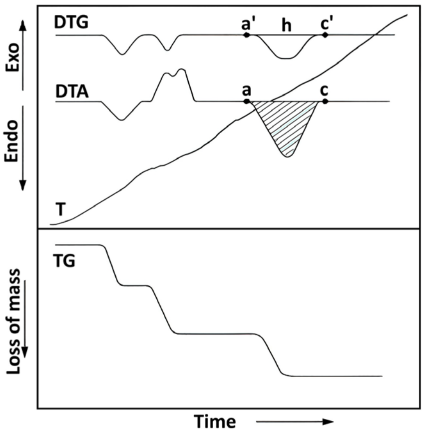

Fig. 2. Example of a derivatogram

During derivatograph analysis, temperature curves (DT) usually show almost linear behavior, except for areas where pronounced endothermic or exothermic processes occur, which lead to local curvatures of the curve.

The DTA curve reflects deviations from the baseline: an increase in the signal corresponds to exothermic phenomena, while a decrease in the signal indicates endothermic processes. Analysis of these changes makes it possible to estimate the quantitative content of the active phase of the material undergoing transformation by determining the enthalpy effects (ΔH) and the temperature difference (ΔT) between the sample and the inert standard (Al_2O_3).

The rate of the irreversible heterogeneous process, accompanied by the formation of gaseous combustion products, which are removed from the reaction zone, is described by the following kinetic equation (1) [18]

$$\frac{dW}{dt}\frac{1}{W_k} = k_0 e^{-E/RT}\left(\frac{W}{W_k}\right)^n, \tag{1}$$

where W_k – mass loss of the test sample; W – mass loss of the test sample at a certain time τ; k_0 – pre-exponential factor; E – activation energy of the process; n – reaction order; T – absolute temperature; R – universal gas constant.

For conditions of linear programmed heating, when $dT / d\tau = q$, equation (1) can be represented in the following form (2)

$$\frac{dW}{dT}\frac{1}{W_k} = \frac{k_0}{q}{}^{-\frac{E}{RT}}\left(\frac{W}{W_k}\right)^n, \tag{2}$$

where q is the heating rate of the test sample.

In this case, the dW / dT value will be proportional to the deviation of the DTG curve from the conditional zero line h (Fig. 2, segment $a'c'$), that is, it will be written in the following form (3)

$$\frac{dW}{dT} = \alpha h. \tag{3}$$

Then the loss of mass of the experimental sample at the initial moment of time can be defined as (4)

$$W_k = k\int_{a'}^{c'}\left(\frac{dW}{dT}\right)\mathrm{d}T = kF. \tag{4}$$

And the mass loss of the test sample up to a certain time point τ for which the comparison is made – in the following form (5)

$$W = k\int_{a}^{c}\left(\frac{dW}{dT}\right)\mathrm{d}T = k\left(F - f\right), \tag{5}$$

where α, k are conversion factors; F is the area under the DTG curve, bounded by the zero line h; f is the area under the DTG curve at a certain time point τ for which the comparison is made.

The ratio of F / f values corresponds to the ratio of the masses of the samples weighed on an analytical balance.

The TG curve reflects the change in the mass of the sample during heating and has a characteristic step shape, which provides an accurate determination of the mass loss of the substance. For the correct interpretation of the TG curve, it is necessary to take into account the mass of the active substance and the upper limit of the measurement range of the instrument. For example, if the mass of the sample and the maximum scale of the instrument are 500 mg, then by determining the height of the corresponding step on the curve and comparing it with the maximum scale, it is possible to calculate the fraction of the mass lost during dehydration or thermal decomposition. The mass loss (%) is determined using an auxiliary grid. The temperature of the onset of thermal destruction (T_p) and the temperature ranges corresponding to the mass loss of the sample are also established.

The DTG curve allows for more accurate localization of the beginning and end of the mass change processes (Fig. 2, points a' and c') compared to the TG or DTA curves. To determine the temperatures corresponding to individual points on the DTA, TG and DTG curves, vertical lines are projected from the selected points to the intersection with the temperature curve *T*.

The complex thermal characterization of wood biomass was carried out using the Q-1500 D experimental equipment, which is part of the Paulik-Erdey thermal analysis system. The derivatograph used in the study (Fig. 3) consists of several functional modules: a power supply, a programmer for adjusting the rate of change of the sample temperature, a thermal block, a weighing module, and a six-channel recorder, of which four channels are used in the work.

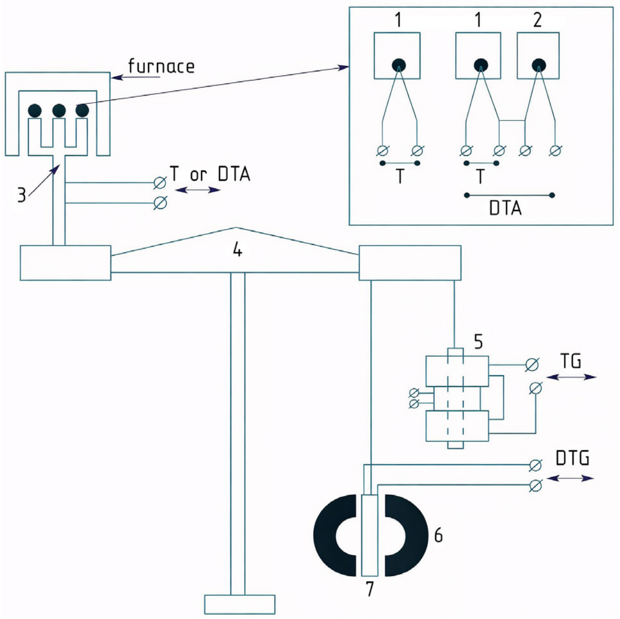

Fig. 3. Schematic diagram of the Q-1500 D derivatograph: 1 — crucibles with samples; 2 — reference sample; 3 — ceramic holder; 4 — analytical thermobalance; 5 — differential transformer; 6 — permanent magnet; 7 — induction coil

The processing of experimental data of thermal analysis was carried out on the basis of the results of thermogravimetric (TG), differential thermogravimetric (DTG) and differential thermal (DTA) curves obtained during the programmed linear heating of wood biomass samples. The primary experimental data recorded by the Q-1500 D derivatograph recorder were digitized and transferred to the Mathcad environment (USA) for further mathematical treatment.

At the first stage, the baselines of the TG, DTG, and DTA curves were aligned in order to eliminate systematic deviations of the baseline caused by the instability of the measuring equipment and zero level errors. For this purpose, data from an inert reference sample (Al_2O_3) were used, which allowed us to correct temperature and mass deviations not related to thermal transformations of biomass.

Next, the characteristic temperature points of the thermal degradation process were determined: the temperature of the onset of decomposition, the temperatures of the maximum mass loss rate, as well as the temperatures of completion of individual stages of thermal transformation. The specified parameters were established by analyzing the extrema of the DTG curves and the corresponding deviations on the DTA curves by projecting the characteristic points onto the temperature curve *T*.

The quantitative assessment of mass loss was carried out using the TG curves by determining the relative change in the mass of the sample in the corresponding temperature intervals. For this purpose, an auxiliary scale of the device was used, taking into account the initial mass of the active substance and the upper limit of measurements. The relative mass loss (%) was determined for each stage of thermal degradation separately, which allowed us to identify the processes of dehydration, depolymerization, and oxidative decomposition of the organic component of wood.

To analyze the kinetic characteristics of the thermal degradation process, DTG curves were used, which determined the mass loss rate dW/dT as a function of temperature. The integration of the areas under the DTG curves was performed to calculate the total and partial mass loss of the sample at a given time or temperature. The obtained values were used to calculate the degree of conversion and further determine the kinetic parameters according to equations (1) to (5).

Enthalpy effects of thermal processes were determined by the area of peaks on DTA curves taking into account the calibration coefficients of the device and the temperature difference between the test sample and the standard. The direction and intensity of the peaks were used to identify endothermic and exothermic stages of thermal transformation of wood biomass.

To increase the reliability of the results, each experimental study was carried out in at least three iterations. The final values of kinetic and thermal parameters were determined as arithmetic means; the errors were estimated by standard deviation. The obtained processed data were used as input parameters for further mathematical modeling of the processes of thermal decomposition and combustion of wood biomass of energy willow *Salix fragilis*.

As part of the experiment, elemental analysis of wood samples was also carried out by the method of direct heating using indicators of the presence of major chemical elements. To determine the elemental composition of wood biomass, current standardized methodologies were used: the content of total sulfur [20], oxygen and nitrogen [21], carbon and hydrogen [22].

The fractional composition of wood biomass was determined by sieve analysis with forced dispersion of the material. The sizes of the sieve openings ranged from 20 to 5000 μm. The quantitative characteristic of the particle residue on the sieves is the fraction of material retained on each sieve with different mesh sizes. Notation: the total particle residue on the sieve with mesh size X – R_X, and the part passing through this sieve is D. For the specified conditions, the following relationship (6) is valid

$$R_X + D = 100\%. \qquad (6)$$

Based on the results of sieve analysis, carried out using a wide range of sieves, the fractional parameters n and b are determined, which characterize, respectively, the degree of heterogeneity of the particle size distribution and the level of fineness of their grinding. These parameters can be determined graphically – according to the dependence of the fractional residue curve $R_X = f(x)$, or analytically – by solving a system of equations constructed by the least squares method:

$$n = \frac{N\sum_{i=1}^{N} Y_i \ln(X_i) - \sum_{i=1}^{N} Y_i \cdot \sum_{i=1}^{N} \ln(X_i)}{N\sum_{i=1}^{N} \ln^2(X_i) + \left[\sum_{i=1}^{N} \ln(X_i)\right]^2}, \qquad (7)$$

$$b = \exp\left(\frac{\sum_{i=1}^{N} Y_i - n\cdot\sum_{i=1}^{N}\ln\left(X_i\right)}{N}\right), \qquad (8)$$

where N is the number of sieves; $Y_i = \ln(100/R_i)$; X_i is the sieve cell size.

During experimental studies aimed at determining the kinetic characteristics of energy biomass, special attention was paid to minimizing the total impact of errors on the calculated parameters. To increase the reliability of the obtained data, an approach was used that reduces the impact of both systematic and random errors.

The use of statistical processing methods of results in combination with an a priori assessment of the accuracy of instruments and experimental procedures allowed us to ensure a high level of data reliability and their compliance with the requirements for further mathematical modeling of biomass combustion processes.

5. Results of investigating the elemental composition and regularities of thermogravimetric decomposition of crushed wood biomass

5. 1. Results of investigating the characteristics of the elemental and fractional composition of experimental samples of wood biomass

In general, the complex thermal characteristics of 70 samples of *Salix fragilis* wood of different ages and dispersed composition of 10 samples per 1 sample of each type were studied.

The averaged experimental technical characteristics of the elemental composition of *Salix fragilis* willow of different ages obtained according to the above-described method, which are decisive for the accuracy and informativeness of the obtained thermal characteristics, are given in Table 1. The fractional composition of the willow samples is given in Table 2, which in the first stage of the series of experiments, in which the influence of exclusively the age characteristics of wood biomass was determined, was kept the same.

Table 1

Elemental composition of wood samples

No. of entry	ID of wood biomass sample	C, %	H, %	S, %	O, %	N, %
1	Willow (1 year)	47.37	4.21	0.00	13.74	0.61
2	Willow (2 years)	51.63	4.59	0.00	10.27	0.52
3	Willow (3 years)	55.88	4.78	0.00	8.16	0.39

Table 2

Fractional composition of wood samples

No. of entry	ID of wood biomass sample	Residue on sieves, R_X, %			
		10 mm	5 mm	2 mm	1 mm
1	Willow (1 year)	7.8	25.1	62.3	4.8
2	Willow (2 years)	7.9	25.2	62.1	4.8
3	Willow (3 years)	7.6	25.5	62.0	4.9

The averaged experimental technical characteristics of the elemental composition of *Salix fragilis* willow samples of different ages and dispersion obtained according to the above-described methodology, which are decisive for the accuracy and informativeness of the obtained thermal characteristics, are given in Table 3. The fractional composition of willow samples is given in Table 4 for the second stage of the series of experiments, in which the influence of age and fractional characteristics of wood biomass was determined.

Table 3

Elemental composition of experimental wood samples

No. of entry	ID of wood biomass sample	C, %	H, %	S, %	O, %	N, %
1	Willow (1-year fine-dispersed)	47.34	4.26	0.00	13.76	0.60
2	Willow (1-year coarse-dispersed)	47.34	4.26	0.00	13.76	0.60
3	Willow (3-year fine-dispersed)	55.83	4.79	0.00	8.12	0.41
4	Willow (3-year coarse-dispersed)	55.83	4.79	0.00	8.12	0.41

Table 4

Fractional composition of experimental wood samples

No. of entry	ID of wood biomass sample	Residue on sieves, R_X, %			
		10 mm	5 mm	2 mm	1 mm
1	Willow (1-year fine-dispersed)	2.9	6.5	65.2	25.4
2	Willow (1-year coarse-dispersed)	7.8	25.1	62.3	4.8
3	Willow (3-year fine-dispersed)	3.0	6.3	65.4	25.3
4	Willow (3-year coarse-dispersed)	7.6	25.5	62.0	4.9

Our research results confirmed the presence of clearly expressed age-related patterns of the elemental composition of woody biomass of *Salix fragilis*. It was established that with increasing age of wood, the content of carbon and hydrogen increases while the content of oxygen and nitrogen decreases, which leads to an increase in the energy value of biomass and the intensity of its thermochemical transformations. It was shown that with the same fractional composition, the influence of age characteristics is a determining factor in the change in the elemental composition. The obtained data confirm the representativeness of the formed samples and lay a reliable basis for further analysis of the kinetic characteristics of thermal destruction and combustion of woody biomass depending on age and dispersion.

5. 2. Results of investigating the thermogravimetric decomposition of crushed biomass of different ages and fractional composition

The experimental methodology included both simple and differential thermometric analysis, as well as thermogravimetric research, which allowed for a comprehensive characterization of the behavior of biomass samples under controlled thermal load.

The thermograms of the experimental samples of the first series of experiments, in which the influence of the age characteristics of wood biomass was determined exclusively, are shown in Fig. 4–6.

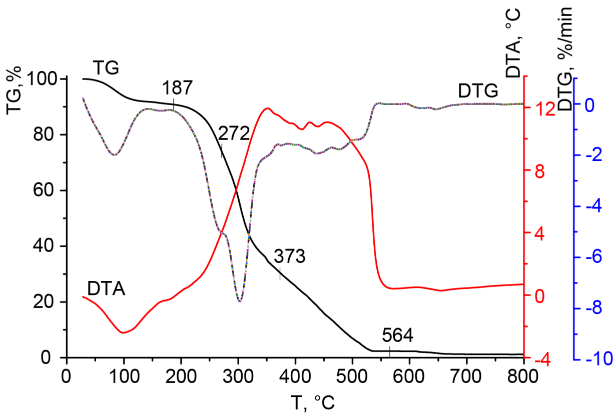

Fig. 4. Thermogram of a sample of 1-year-old willow wood (sample 1)

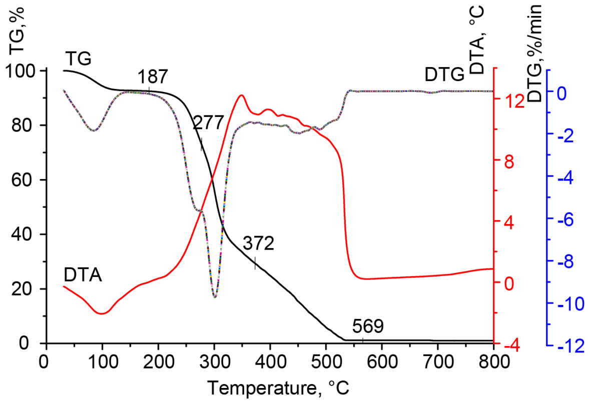

Fig. 5. Thermogram of a sample of two-year-old willow wood (sample 2)

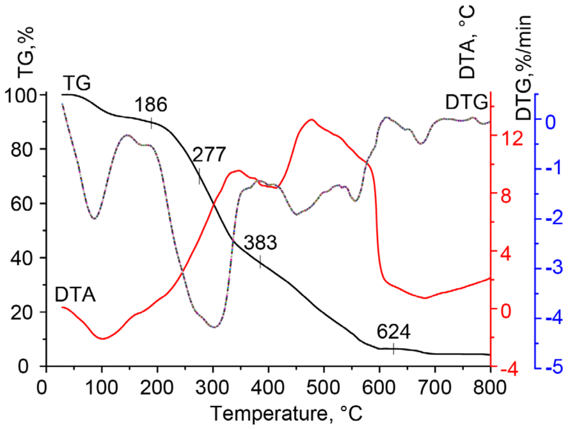

Fig. 6. Thermogram of a sample of three-year-old willow wood (sample 3)

The results of processing thermograms by differential thermogravimetry and differential thermal analysis of experimental samples are given in Table 5.

Fig. 7, 8 show a comparison of TG and DTA curves of *Salix fragilis* willow wood samples.

In order to qualitatively assess the influence of not only the age but also the degree of dispersion (fractional composition of samples) of the energy willow *Salix fragilis* on the features of its thermal decomposition, a study of four types of samples was conducted. The sample included samples of one-year-old willow with a fine-dispersed structure (sample 1) and a coarse-dispersed fraction (sample 2), as well as three-year-old willow with a fine-dispersed structure (sample 3) and coarse-dispersed (sample 4), respectively.

Table 5

Results of complex thermogravimetric and differential thermogravimetric analysis of *Salix fragilis* willow samples

Sample	Stage	Temperature interval, °C	Mass loss, %
1-year-old willow (sample 1)	1	21–188	7.7
	2	186–276	19.6
	3	275–373	43.4
	4	371–568	28.6
2-year-old willow (sample 2)	1	20–188	9.1
	2	186–273	17.4
	3	273–372	42.8
	4	372–565	28.2
3-year-old willow (sample 3)	1	21–185	10.1
	2	185–278	20.3
	3	276–384	31.3
	4	384–625	32.0

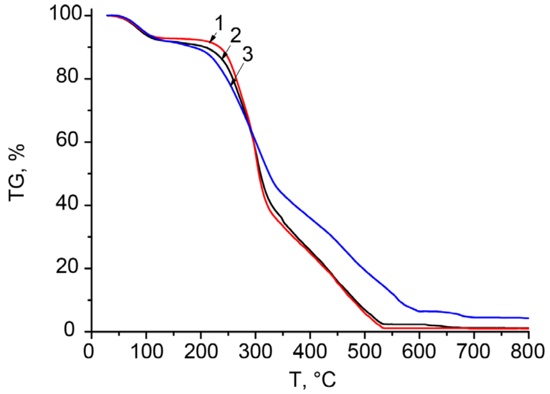

Fig. 7. Thermogravimetric study curves:
1 — sample 1; 2 — sample 2; 3 — sample 3

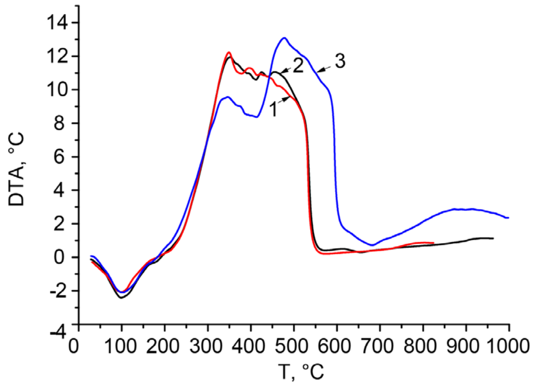

Fig. 8. Thermogravimetric study curves:
1 — sample 1; 2 — sample 2; 3 — sample 3

The results of thermogravimetric analysis of the specified samples of the second series of experiments, in which not only the influence of age but also fractional characteristics of wood biomass was determined, are given in Table 6.

Table 6

Results of complex thermogravimetric and differential thermogravimetric analysis of *Salix fragilis* willow samples

Sample	Stage	Temperature interval, °C	Mass loss, %
1-year-old finely dispersed willow (sample 1)	1	21–169	9.86
	2	173–268	20.02
	3	271–413	39.13
	4	416–505	30.93
1-year-old large-dispersed willow (sample 2)	1	21–171	8.17
	2	172–276	17.04
	3	274–422	49.25
	4	422–531	25.53
Three-year-old finely dispersed willow (sample 3)	1	20–171	9.86
	2	171–271	20.31
	3	272–416	40.24
	4	415–515	29.58
Three-year-old coarsely-dispersed willow (sample 4)	1	21–174	8.17
	2	170–278	17.02
	3	276–430	49.24
	4	423–552	25.55

Thermograms of experimental samples of the second series of experiments, in which not only the influence of age but also fractional characteristics of wood biomass were determined, are shown in Fig. 9, 10.

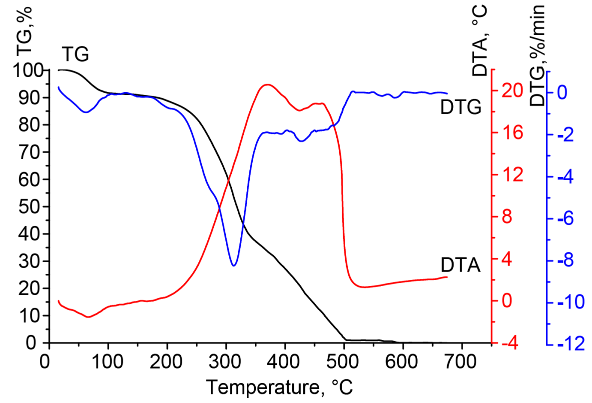

Fig. 9. Thermogram of a sample of fine-grained 1-year-old willow wood (sample 1)

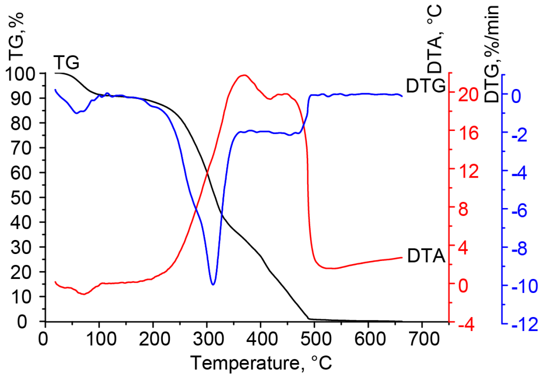

Fig. 10. Thermogram of a sample of three-year-old fine-grained willow wood (sample 3)

Fig. 11–13 illustrate a comparative analysis of thermogravimetric (TG) and differential thermal analysis (DTA) curves for different types of samples.

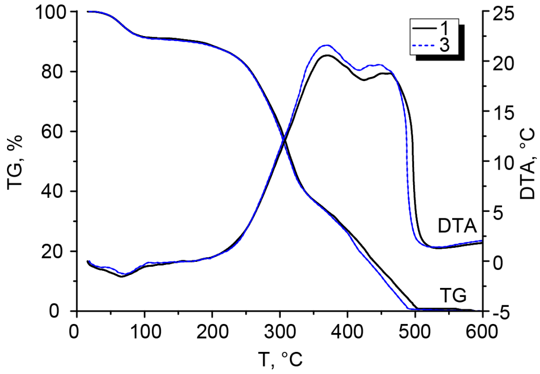

Fig. 11. Comparison of thermogravimetric and differential thermoanalytical curves of samples 1 and 3

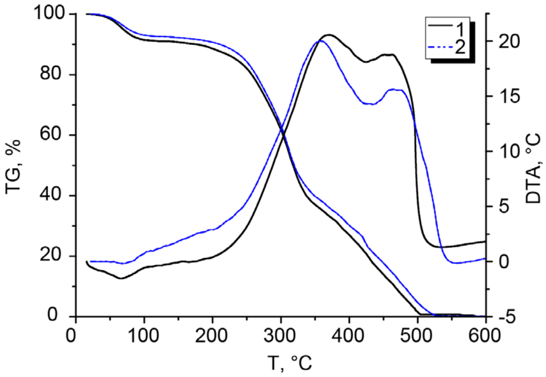

Fig. 12. Comparison of thermogravimetric and differential thermoanalytical curves of samples 1 and 2

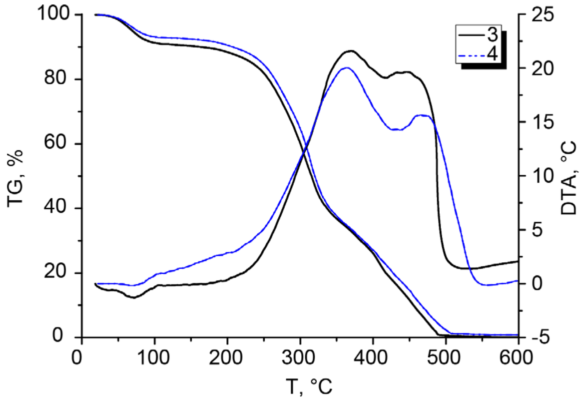

Fig. 13. Comparison of thermogravimetric and differential thermoanalytical curves of samples 3 and 4

Our complex thermogravimetric and differential thermal analyses revealed that the thermal decomposition of the crushed biomass of *Salix fragilis* has a clearly pronounced staged character, and the temperature intervals and the intensity of mass losses significantly depend on both the age of the wood and its fractional composition. It was established that with increasing biomass age, the proportion of mass losses at the stages of active thermo-oxidative destruction and burning of the carbonized residue increases, which correlates with the increased carbon content. It was shown that fine-dispersed samples are characterized by an earlier onset and higher intensity of destructive processes due to the increased specific surface area and the release of volatile substances, while in coarse-dispersed fractions thermolysis proceeds more slowly with the expansion of the temperature intervals of individual stages. Our results confirm the decisive influence of age and fractional characteristics on the kinetics of thermal destruction of wood biomass and lay a scientifically sound basis for further determination of the kinetic parameters of its combustion in real heat engineering systems.

6. Discussion of results based on investigating the characteristics of the composition and thermogravimetric decomposition of crushed samples of wood biomass

Analysis of the elemental composition of wood biomass *Salix fragilis* revealed clear age-related patterns: with increasing age of trees, the content of carbon and hydrogen increases, which increases the energy value of wood, while the proportion of oxygen and nitrogen decreases. Such changes indicate a decrease in the relative amount of volatile components and simultaneously affect the ash content and chemical stability of biomass, which is confirmed by data in Tables 1, 3. Fractional analysis of samples revealed that the degree of dispersion significantly determines the uniformity of particle distribution and, as a result, the intensity of thermooxidative processes (Tables 4, 6). Elimination of variability of the fractional composition at the first stage allowed us to determine the direct effect of wood age on the energy and kinetic characteristics of thermolysis (Fig. 4–8).

Comparison with the data reported in [23] confirms similar patterns: the cited work shows that the increased carbon and hydrogen content in older wood samples contributes to a more intensive release of volatile components during thermal decomposition, which is consistent with our observations on three-year-old *Salix fragilis* samples. In addition, the dispersion of the material in both studies affects the rate of thermolysis, which confirms the universality of the influence of the physical structure of wood on the kinetics of thermooxidative processes. Thermograms of experimental samples of the first series of experiments, in which the influence of age characteristics of wood biomass was determined exclusively, are shown in Fig. 4–6. The averaged thermal analysis of the crushed biomass of *Salix fragilis* willow (Fig. 4–6) allowed us to distinguish four consecutive stages of thermolysis, each of which is characterized by specific physicochemical transformations, mass loss, and characteristic thermogravimetric and differential thermal signals.

The first stage (21–186°C) corresponds to endothermic processes associated with the evaporation of surface moisture, as well as partial release of capillary moisture. At this stage, a gradual loss of mass is observed, which increases when the temperature reaches more than 200°C, which is clearly recorded on the TG curves.

The second stage (186–276°C), according to the results of differential thermogravimetric analysis, includes both endothermic processes of dehydration and pyrolysis (elimination of volatile decomposition products), and the development of exothermic thermooxidative reactions. These reactions are accompanied by a sharp decrease in the degree of cellulose polymerization, as evidenced by the appearance of clearly expressed peaks on the DTA curves of *Salix fragilis* samples (Fig. 4–6). Sample 3 demonstrates the most intense loss of mass at this stage, which indicates a more active elimination of volatile products from the wood bark.

The third stage (276–379°C) is characterized by the most intense mass loss, which is confirmed by the data on TG analysis (Table 5). At this stage, active thermo-oxidative destructive processes occur with flame combustion of volatile components. DTA curves record intense exothermic effects. In the case of sample 3, the exothermic activity is the lowest among all samples, which indicates less intense combustion in the air phase. The fourth stage (379–625°C) covers the burnout of the carbonized residue. For sample 3, which demonstrates the highest content of coke residue, the most intense course of the heterogeneous oxidation process is noted. The corresponding exothermic peak on the DTA curve is the most pronounced and shifts to the higher temperature zone compared to other samples.

Comparison with study [23] confirms our observed regularities: in the cited work, it is shown that older wood samples with higher carbon and hydrogen content demonstrate more intensive release of volatile components during pyrolysis, as well as enhanced combustion of residual carbonized mass. This is consistent with our observations for three-year-old *Salix fragilis* samples, in which the maximum exothermic DTA peaks and greater mass loss reflect active thermo-oxidative processes at the third and fourth stages of thermolysis.

During the second series of experiments, not only the influence of age but also fractional characteristics of wood biomass was determined. Fig. 9, 10 show thermograms of finely dispersed samples of one- and three-year-old *Salix fragilis* wood. The first stage of thermolysis of samples 1 and 3, which occurs in the temperature range of 21–171°C, is characterized by endothermic processes associated with the evaporation of surface and capillary moisture, with a mass loss of about 9.9%.

At the second stage of thermolysis, in the range of 171–269°C, endothermic reactions of dehydration and pyrolysis occur simultaneously, accompanied by the release of volatile products of thermal destruction and a decrease in the degree of cellulose polymerization (mass loss is approximately 20.2%). Exothermic thermooxidative reactions occur in parallel, which is reflected in the DTA curves of the studied samples.

The third stage of thermolysis, in the range of 272–414°C, is characterized by maximum mass loss and a pronounced exothermic effect on the DTA curves. This corresponds to active processes of thermal-oxidative destruction and flame combustion of volatile products. For sample 1, the mass loss at this stage is 39.13%, while for sample 3 it is 40.24%.

The fourth stage, in the range of 415–510°C for sample 1 and 415–500°C for sample 3, corresponds to the final burnout of the carbonized residue, with a mass loss of 30.93% for sample 1 and 29.58% for sample 3. In the three-year-old sample (sample 3), the processes of flame combustion and oxidation of the carbonized residue are more intense, which is confirmed by a greater mass loss and more pronounced exothermic peaks on the DTA curves (Fig. 11). These differences are explained by the increased carbon content in the three-year-old samples of 55.83% (Table 4), which contributes to a more active thermooxidative reactivity in the indicated temperature ranges. Similarly, the authors of [24] note the decisive role of the carbon content and structural organization of wood in the formation of the kinetic parameters of pyrolysis.

Fig. 12, 13 show a comparison of thermogravimetric (TG) and differential thermoanalytical (DTA) curves of samples of different dispersion. Thermal decomposition of coarse-dispersed samples occurs more slowly compared to fine-dispersed ones, which is confirmed by a smaller mass loss at all stages and weaker thermal effects on DTA curves. A similar effect of dispersion on the rate of thermal destruction was recorded in works [25–27], in which it was shown that a decrease in particle size increases the intensity of flame combustion and the completeness of burnout. At the same time, unlike the above papers, in our study the effect of age characteristics and fractional composition was considered comprehensively for one type of fast-growing energy crop, which allowed us to identify specific patterns specifically for *Salix fragilis*.

This effect can be explained by the influence of the specific surface: with a decrease in particle size, the area of the contact surface with air increases, which contributes to a faster course of destructive, thermo-oxidative and heterogeneous oxidation processes.

In general, the results of thermogravimetric and differential thermal analysis indicate that three-year-old willow wood *Salix fragilis* has the highest calorific value. This is confirmed by the maximum exothermic effects on DTA curves, associated with the intensive course of thermo-oxidative destruction and the burning of residual organic mass.

The main limitations of our study include the laboratory nature of the experiments and the use of controlled heating conditions, which do not fully reproduce the real operating modes of industrial boilers. The obtained kinetic characteristics are correct within the studied temperature ranges, fractional composition, and heating rates, and require adaptation when extrapolating to other types of heat-technical equipment or variable combustion conditions.

The disadvantage of the work is the lack of direct determination of kinetic constants of individual stages of thermolysis and the limited number of heating rates, which complicates the construction of a generalized kinetic model. In the future, these shortcomings can be eliminated by using isoconversion methods of analysis, expanding the range of experimental conditions and combining thermal analysis with gas analysis of decomposition products.

Further development of our study should be directed to determining the kinetic parameters of thermal destruction of *Salix fragilis* using differential and integral methods, as well as to validating the results obtained at semi-industrial and industrial installations. The main difficulties at this stage may be associated with the complication of the mathematical description of multi-stage processes, the need to scale the results and ensure the reproducibility of experiments under conditions of variable biomass composition.

7. Conclusions

1. We have found that the elemental and fractional composition of the woody biomass of *Salix fragilis* significantly depend on the age of the plants. With increasing age from one to three years, the carbon content increases from 47.34–47.37% to 55.83–55.88%, hydrogen – from 4.21–4.26% to 4.78–4.79%, while the oxygen content decreases from 13.74–13.76% to 8.12–8.16%. Nitrogen is contained in insignificant quantities (0.39–0.61%), and sulfur is practically absent. An age-related change in the fractional composition was revealed: fine-dispersed samples are characterized by the dominance of fractions of 1–2 mm (62–65%), while in coarse-dispersed samples the bulk falls on fractions of 5–10 mm. The obtained patterns are explained by changes in the structural organization of wood and the accumulation of carbon-containing components with age, which distinguishes energy willow from young woody biomass of other fast-growing crops.

2. It is shown that the thermal destruction of crushed *Salix fragilis* biomass has a clearly pronounced staged nature and includes four consecutive stages in the temperature range of 21–625°C with a total mass loss of up to 95%. At the first stage (21–186°C), endothermic evaporation of physically bound moisture occurs with a mass loss of 7.7–10.1%. The second stage (171–278°C) is characterized by dehydration and initial pyrolysis with partial release of volatile components and a mass loss of 17.4–20.3%. The third stage (272–384°C) corresponds to intensive thermo-oxidative processes and flame combustion of volatile substances, which is accompanied by a maximum mass loss of 31.3–43.4%. At the fourth stage (379–625°C), complete burning of the carbonized residue occurs with an additional mass loss of 28.2–32.0%. It was found that three-year-old samples are characterized by higher exothermic activity and greater mass loss at the stages of pyrolysis and flame combustion (up to 43.4%) compared to one-year-old samples (up to 39.1%). Finely dispersed biomass demonstrates more intensive thermo-oxidative processes due to a larger specific surface, while in coarsely dispersed samples the destruction occurs more slowly with a shift in temperature peaks. The identified features are explained by the combined influence of age-related changes in elemental composition and dispersity, which determines the kinetics of heat and mass transfer processes during thermolysis.

Conflicts of interest

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study, as well as the results reported in this paper.

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Data availability

All data are available, either in numerical or graphical form, in the main text of the manuscript.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

Authors' contributions

Andrii Kapustianskyi: Conceptualization, Methodology, Writing – review & editing, Formal analysis; **Kateryna Romanova**: Writing – original draft, Validation, **Igor Galyanchuk**: Validation, Visualization; **Oksana Yurasova**: Validation, Visualization.

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