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STUDY OF THE KINETICS OF THERMAL DECOMPOSITION OF LINDEN WOOD USING THERMOGRAVIMETRIC ANALYSIS IN AN AIR ATMOSPHERE

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Abstract

The article investigates the kinetics of thermal decomposition of linden wood using TGA in air at a heating rate of 10–30 °C/min. Temperature ranges and mass loss were determined for the stages of dehydration, pyrolysis, and combustion of the carbon residue. It is shown that biomass decomposition occurs through the decomposition of hemicellulose, cellulose, and lignin, that constitute its composition. The influence of the heating rate on the temperature of maximum mass loss, which ranges from 350 °C to 370 °C corresponding to cellulose decomposition, was studied. Kinetic parameters of pyrolysis and for individual stages of hemicellulose and cellulose decomposition were calculated using the Coats-Redfern method.

Key words: biomass, linden wood, pyrolysis, volatiles, coke residue, TGA, chemical kinetics, reaction rate constant, activation energy.

Replacing fossil fuels with renewable alternatives, including those derived from biomass, is one of the key elements of many national decarbonization strategies. The draft National Action Plan for the Development of Renewable Energy up to 2030 sets an indicative target to triple the share of energy from renewable sources in final energy consumption – from 9% in 2020 to 27% in 2030, including reaching 25% in the electricity sector. Additionally, substituting scarce organic fuels, primarily coal and gas, during the martial law period at industrial facilities with renewables is **extremely relevant**. It should be noted that the Russian invasion of Ukraine has caused massive disruption of economic activity links and destruction of the fuel and energy infrastructure of Ukraine. According to the Recovery and Development Plan of Ukraine proposed by the government and president of Ukraine, the development of renewable energy is of key importance for shaping the future structure of Ukraine's power generation capacities. The implementation of this plan is a chance to restore the war-inflicted damages. The growth of renewable fuels usage, including various types of biomasses, is envisaged in Ukraine's Energy Strategy through 2035.

Waste-to-energy (WTE) technologies are rapidly developing today, offering the potential to generate renewable energy from biomass, including agricultural and processing industry wastes [1]. The improvement of existing and development of new WTE technological systems for burning biomass, including wood-processing industry waste, should be based on an understanding of the

patterns of thermal processing of such fuels [2]. **The aim of this work** is to study the patterns of thermal decomposition of biomass samples.

Significant attention in the design of furnace devices is devoted to the process of combustion of particles of the original fuel. The burning of solid organic fuel is a complex physico-chemical process, which is conventionally divided into several main stages. The first stage is the pyrolysis of the fuel particle, which includes its heating, drying, formation, and release of volatiles. The second stage is the combustion of volatiles and the coke residue, including their ignition. These stages differ in speed, duration, and impact on the combustion process depending on technological conditions [3, 4].

Pyrolysis is a process of thermal decomposition of the fuel, resulting in the formation (synthesis) of volatile substances (mainly H_2 , CH_4 , CO , CO_2 , smaller amounts of C_2H_6 , C_2H_4 , high-gas organic compounds, and water vapor), solid coke residue, and liquid products (resins) [5, 6]. This process involves multiple phases, complex chemical pathways, unstable intermediate products, and the effects of heat and mass transfer. The quantitative and qualitative composition of the products formed depends on the type of fuel, final pyrolysis temperature, heating rate and particle size, reactor pressure, residence time of particles in the reactor, conditions of volatiles evacuation, and the composition of the surrounding gas environment.

All types of biomass are fuels with a high volatile content (about 80–85% on a dry, ash-free basis), which is why pyrolysis is the most important process for the thermal treatment of such fuels. The yield of volatile substances can significantly affect the subsequent ignition of the coke residue. With a high yield of volatiles, the combustion process proceeds faster, and their gaseous and liquid contents influence the burning rate. The heating rate of the fuel during pyrolysis determines the ratio of volatile output to coke residue.

The following stages of pyrolysis of the solid fuel are distinguished: breaking of the weakest intramolecular bonds, intramolecular rearrangements, release of sorbed gases (at temperatures up to 200 °C); absorption of energy to enhance thermal vibrations of molecules (200–350 °C); predominance of decomposition reactions, change in the aggregate state of the coal substance (350–450 °C); release of tars, hardening of the plastic mass, dominance of synthesis reactions (450–550 °C); combination of large radical fragments in the solid phase, intensive gas release of vapors and tarry substances (> 550 °C) [3, 4].

During slow heating, the molecular bonds of the organic substance break sequentially, starting from the weakest. The typical order of volatile release is as follows: first, the release of sorbed gases occurs, followed by the destruction and synthesis of water vapor and CO_2 , then CH_4 , CO , aliphatic hydrocarbons, tars, and H_2 . The combustion of coke residue is generally the longest stage and is accompanied by the interaction of oxygen O_2 , carbon dioxide CO_2 , water vapor H_2O , and to a lesser extent hydrogen H_2 with the external and internal surface of the particle. The primary products of the heterogeneous interaction of carbon C with O_2 are both CO and CO_2 , and the overall reaction is represented as: $(1+m) \cdot C + (m/2+1) \cdot O_2 \rightarrow m \cdot CO + CO_2$, where m characterizes the completeness of carbon combustion to CO_2 : at $m = 0$ – the reaction product is only CO_2 ; at $m \gg 1$ – CO .

The ratio of primary CO/CO_2 increases with rising temperature and decreasing pressure and can be represented in an Arrhenius form: $m = CO/CO_2 = A \exp(-E_a/RT)$, where A is a constant determined experimentally; E_a is the activation energy, J/mol; $R = 8.31$ kJ/(kmol · K) – is the universal gas constant; T is the temperature, K.

The main reactions of oxygen interaction with carbon occurring on the particle and pore surfaces are: $C + O_2 \rightarrow CO_2 + 395$ kJ/mol, $2C + O_2 \rightarrow 2CO + 219$ kJ/mol. It is considered that oxygen interaction does not occur over the entire accessible surface, but only at certain regions – active sites, whose presence is determined by carbon edges or dislocations, inorganic impurities, oxygen- or hydrogen-containing functional groups. The successive elementary stages of the reaction are:

physical and chemical adsorption of the gas, migration of the intermediate complex, desorption of reaction products. With a sufficient amount of oxygen in the gas phase, oxidation of pyrolysis products occurs: $2\text{CO} + \text{O}_2 \rightarrow 2\text{CO}_2 + 571 \text{ kJ/mol}$, $2\text{H}_2 + \text{O}_2 \rightarrow 2\text{H}_2\text{O} + 231 \text{ kJ/mol}$, $\text{CH}_4 + 2\text{O}_2 \rightarrow \text{CO}_2 + 2\text{H}_2\text{O} + 892 \text{ kJ/mol}$. The specified reactions proceed with significant heat release. Having different rates, they affect the combustion process differently. A distinctive feature of the $\text{CO} + \text{O}_2$ reaction is its sensitivity to water vapor. "Dry" oxidation of CO (in the absence of water vapor) is characterized by an extremely low reaction rate. "Wet" oxidation occurs much faster and is controlled by the following elementary step: $\text{CO} + \text{OH} \rightarrow \text{CO}_2 + \text{H}$.

Biomass is a highly complex fuel in terms of composition, whose main organic components are cellulose, hemicellulose, and lignin [6]. Cellulose is a glucose polysaccharide with the formula $\text{C}_6\text{H}_{10}\text{O}_5$, featuring a cyclic structure with high stability due to hydrogen bonds [7]. Hemicellulose is a heteropolysaccharide, often represented as $(\text{C}_5\text{H}_8\text{O}_4)_n$. It is an amorphous, thermally softening organic polymer that decomposes quickly at moderate temperatures and consists of various monosaccharides such as xylan [6]. Lignin is a large, hydrophobic, aromatic polymer with phenolic linkages, having the formula $[\text{C}_9\text{H}_{10}\text{O}_3(\text{OCH}_3)_{0.9 \dots 1.7}]_n$. It provides structural support to cellulose and hemicellulose within plant cells [8]. Overall, cellulose makes up 35–55% of biomass, hemicellulose 23%–32%, and lignin 15%–25% [9]. The content of other components, specifically inorganic and extractive substances, accounts for 5–13% [10]. The content of each component varies depending on the type of biomass and its part, for example, stems or sunflower husks, which affects their combustion temperature and chemical composition [11]. In woody biomass, cellulose content ranges between 40% and 60%, while lignin content varies from 21% to 30% [12].

Thermal decomposition of biomass occurs as a result of the breakdown of polymer chains (cellulose, lignin, hemicelluloses) that make up its structure. The process of thermal degradation of cellulose begins at temperatures above 240 °C and is fully completed at around 450 °C [5, 12]. Hemicelluloses thermally degrade within the temperature range of 200 °C to 260 °C. Thermal degradation of lignin starts at 200 °C, and between 225 °C and 450 °C, the reaction process becomes exothermic. Some additional studies have shown that lignin reaction begins at low temperatures, but pyrolysis can continue to higher temperatures [12, 13]. Thermal degradation of lignin produces 1.5 times more coke than cellulose [12]. Pyrolysis in wood typically starts at 200 °C and continues up to 450–500 °C, depending on the wood species. The actual reaction scheme of wood pyrolysis is extremely complex due to the formation of more than a hundred intermediate products.

Materials and methods

The experiments studied samples of linden wood chips. The averaged technical analysis of linden wood on a dry basis is as follows: $A^r = 0.3\%–0.5\%$, $V^r = 63\%–68\%$, $W^r = 20\%$, fixed carbon $C_f^r = 11\%–14\%$. The elemental composition of the fuel is $C^r = 39\%–41\%$, $O^r = 33\%–35\%$, $H^r = 5.5\%–6.0\%$, $N^r = 5.5\%–6.0\%$, and $S^r < 0.05\%$. The Lower Heating Value is 14.5–15.2 MJ/kg.

One of the reliable methods for studying the mechanisms of thermal conversion of solid fuels is thermogravimetric analysis (TGA), which allows for the assessment of mass loss, temperature ranges of reactions, and kinetic parameters. Research on the thermal conversion of fuel particle combustion was conducted by interacting their organic component with atmospheric oxygen at atmospheric pressure. The setup is based on a vertical cylindrical furnace with a height of $h = 0.4 \text{ m}$ and an internal diameter of $d = 0.12 \text{ m}$ (see Fig. 1).

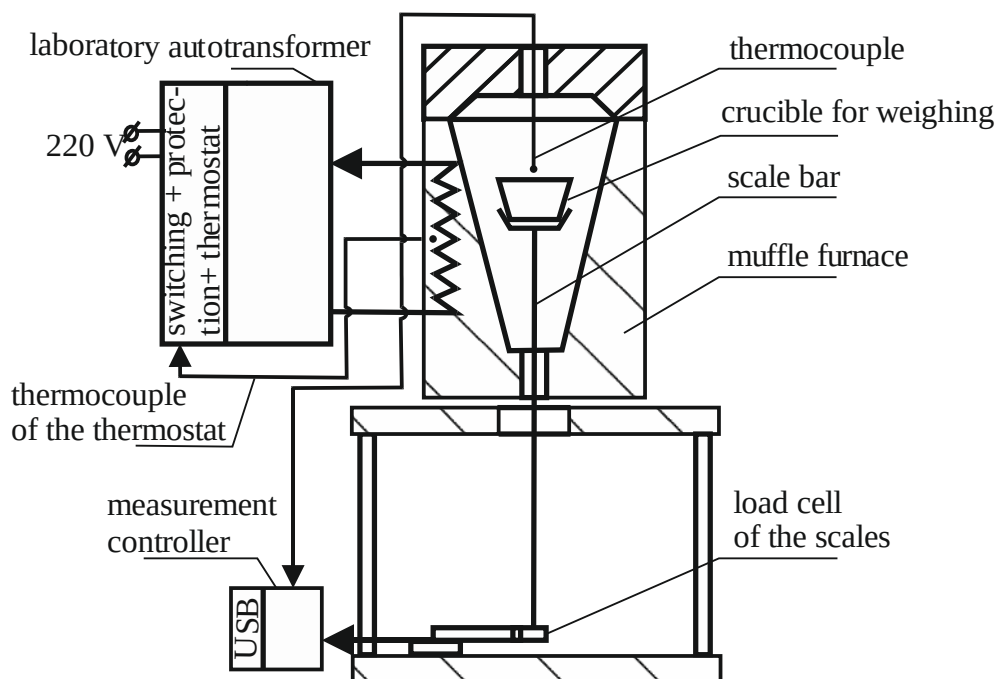


Fig. 1. Schematic diagram of the setup for studying the thermochemical conversion of solid fuel

The operation of the setup is based on the principle of TGA, that is, continuous recording of the change in mass of the solid fuel sample under study during the linear temperature change in the reaction zone under conditions of natural convection of the gas reagent. The maximum furnace operating temperature is up to 1000 °C. Depending on the sample density, samples weighing up to 3–5 g can be studied with an accuracy of 0.001 g.

The procedure for conducting a TGA experiment is as follows. The fuel sample, ground to the required size, is weighed and placed in the cold furnace. After placing the studied fuel charge, the furnace lid is closed and heating is turned on to the temperature set by the thermostat. After reaching temperatures significant for the experiment (depending on the sample material, usually 30–40 °C), recording of temperature T , °C, and mass change m , mg, begins in a file on the PC. The temperature range was from 30 °C to 800 °C. Data recording is stopped after the change in sample mass stabilizes (its burnout). Result: we have dependencies of mass and temperature as a function of time τ , s. The availability of primary data in the form of dependencies $m = f(\tau)$ and $T = g(\tau)$ allows obtaining data in the form of $m = g(T)$, $W = dm(\tau)/d\tau$, i.e., the reaction rate, and many others needed to determine the kinetic/dynamic characteristics according to the selected models. Linden wood samples were studied in an air atmosphere at three heating rates: 10, 20, and 30 °C/min. TGA curves were constructed for each case.

Processing of Experimental Data. TGA data can be analyzed to obtain the kinetic parameters of the thermal decomposition of solids, in our study of linden wood, specifically the activation energy E_a , kJ/mol and pre-exponential factor A , s⁻¹. The degree of conversion at time τ (indicating the fraction of the experimental sample that has reacted by time τ) is determined by the formula $\alpha = \frac{m_0 - m_\tau}{m_0 - m_\infty}$, where

m_0 is the initial mass of the sample; m_∞ is the final mass at the stage of thermal decomposition; m_τ is the current mass of the sample. The change in the degree of fuel conversion α as a function of time τ is described by an Arrhenius-type equation: $\frac{d\alpha}{d\tau} = A \exp\left(-\frac{E_a}{RT}\right) f(\alpha)$, where $f(\alpha)$ is a function characterizing the reaction model [14, 15].

In non-isothermal TGA experiments, the heating rate is also a function of time, expressed in the following form: $\frac{d\alpha}{d\tau} = \frac{d\alpha}{dT} \frac{dT}{d\tau}$. For non-isothermal measurements, the linear heating rate β (°C/s) can be defined as $\beta = dT/d\tau$. It can be written as: $\frac{d\alpha}{d\tau} = \frac{d\alpha}{dT} \beta$, or $\frac{d\alpha}{dT} \beta = A \exp\left(-\frac{E_a}{RT}\right) f(\alpha)$.

This equation is known as the general equation of the TGA curve. We separate the variables and obtain: $\frac{d\alpha}{f(\alpha)} = \frac{A}{\beta} \exp\left(-\frac{E_a}{RT}\right) dT$. We integrate the last equation taking into account that

$g(\alpha) = \int_0^\alpha \frac{d\alpha}{f(\alpha)}$: $g(\alpha) = \frac{A}{\beta} \int_{T_0}^T \exp\left(-\frac{E_a}{RT}\right) dT$. Using the transformations given in [14], it can be written as: $\int_{T_0}^T \exp\left(-\frac{E_a}{RT}\right) dT \approx \frac{R}{E_a} T^2 \exp\left(-\frac{E_a}{RT}\right)$, or $g(\alpha) = \frac{AR}{\beta E_a} T^2 \left(1 - \frac{2RT}{E_a}\right) \exp\left(-\frac{E_a}{RT}\right)$. If the last equation is integrated using the Cotes and Redfern method [14, 15], one can obtain:

$\ln \frac{g(\alpha)}{T^2} = \ln \frac{AR}{\beta E_a} \left(1 - \frac{2RT}{E_a}\right) - \frac{E_a}{RT}$. The magnitude of $2RT/E_a$ is negligible compared to one for the thermal decomposition of lignocellulosic materials, so the plot of $\ln[g(\alpha)/T^2]$ versus $1/T$ will be linear with a slope of $-E_a/R$, since $\ln[AR/\beta E_a]$ remains almost constant. The functions $f(\alpha)$ and $g(\alpha)$ depend on the mathematical model of each transformation mechanism. Using the appropriate $g(\alpha)$, the plot of $\ln[g(\alpha)/T^2]$ versus $1/T$ demonstrates linearity with a high coefficient of determination (R_2) in the regression analysis, allowing for the determination of the activation energy and the pre-exponential factor.

For different reaction orders n and models, $f(\alpha)$ is a tabulated function: for $n = 1$ $f(\alpha) = 1 - \alpha$ [14, 15]. For $n = 1$, we will write the last equation as follows: $\frac{-\ln(1-\alpha)}{T^2} = \frac{AR}{\beta E_a} \exp\left(-\frac{E_a}{RT}\right)$. We logarithmise and get: $\ln\left[\frac{-\ln(1-\alpha)}{T^2}\right] = \ln \frac{AR}{\beta E_a} - \frac{E_a}{RT}$ or $n \neq 1$ $g(\alpha) = \frac{(1-\alpha)^{-(n-1)} - 1}{n-1}$.

Results and discussion

Figure 2 shows the typical dependence of the degree of conversion α on temperature T for different heating rates. The thermodestruction process occurs in several stages.

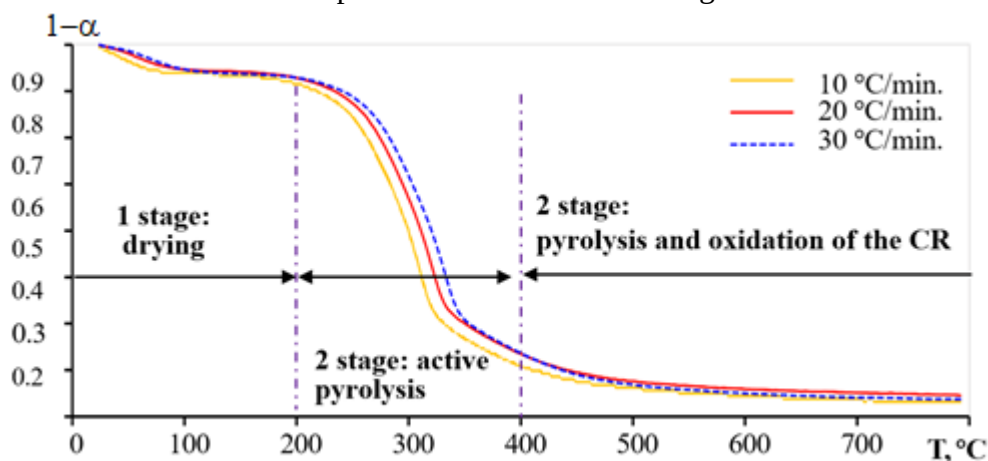


Fig. 2. Typical dependence of the conversion degree α on T for different heating rates

Dehydration (drying) – dehydration of low-molecular-weight components, removal of physically bound water (up to 150–200 °C): mass loss ~ 5%.

Pyrolysis at 200–405 °C – active pyrolysis. Decomposition of hemicellulose (200–280 °C) – release of CO, CO₂, and water vapor; mass loss ~ 10%–15%. Decomposition of cellulose (290–

400 °C) – numerous reactions involving the cleavage of C–C and C–O bonds and radical reactions forming gases or volatile compounds, mainly due to the breakdown of higher carbohydrates; primary stage of volatile formation, tar, and condensed hydrocarbons; mass loss ~ 60%–70%. This correlates with the cellulose content in the wood being up to 60% [12]. Beginning of lignin decomposition.

Pyrolysis and oxidation of the carbon residue (CR). Lignin decomposition (350–550 °C) – slow breakdown into phenols, tars, and gaseous products; mass loss ~10%–15%. Oxidation of CR (> 500 °C): coke residue accounts for about 1%–2% of the initial mass.

Primary decomposition of biomass material starts with the degradation process (< 200 °C), while secondary pyrolysis (> 350–400 °C) includes aromatization processes. The most significant structural changes in biomass occur at 300–400 °C, characterized by a decrease in the intensity of C–O and C=C bonds and the formation of C–C bonds. With increasing temperature, there is a continuous decrease in the intensity of valence –OH bonds and an increase in the yield of aromatic compounds, peaking above 600 °C, which depends on the ratio of cellulose to lignin in the fuel.

With an increase in the heating rate from 10 to 30 °C/min: the pyrolysis temperature range shifts toward higher temperatures; the rate of mass loss increases; less carbon residue is formed due to more intensive oxidation. At the same time, the temperature at the maximum rate of pyrolysis for a heating rate of 10 °C/min is about 350 °C and shifts to higher values (~370 °C) at 30 °C/min.

Kinetic parameters for the main decomposition stage – active pyrolysis (200–405 °C) – have been determined. Results were obtained for $n = 1$: $E_a = 68$ kJ/mol, $A = 2.6 \times 10^3$ s⁻¹ ($R^2 = 0.84$). The activation energy represents the minimum energy required to initiate the decomposition reaction – in this case, it indicates the moderate stability of the wood's organic structure. The reaction order, close to first order, suggests that the decomposition process is independent of the initial substance concentration. Kinetic parameters were also determined for the hemicellulose decomposition stage (200–280 °C) and for the cellulose decomposition stage (290–405 °C). Results were obtained for $n = 1$: $E_a = 26$ kJ/mol, $A = 2.6 \times 10^7$ s⁻¹ ($R^2 = 0.84$) for 200–280 °C and $E_a = 126$ kJ/mol, $A = 3.1 \times 10^8$ s⁻¹ ($R^2 = 0.996$) for 290–405 °C.

Conclusions

The pyrolysis of linden wood takes place in several stages with the predominant thermochemical decomposition in the temperature range of 200–405 °C. An increase in the heating rate from 10 to 30 °C/min leads to a shift in the maximum decomposition to higher temperatures and a decrease in the yield of coke residue.

The kinetic parameters of pyrolysis were calculated for individual stages of hemicellulose and cellulose decomposition using the Coates-Redfern method. The maximum activation energy (126 kJ/mol) and decomposition rate are achieved in the temperature range of 290–405 °C and correspond to the cellulose decomposition stage. The R^2 value for the cellulose decomposition stage is 0.996, indicating that the pyrolysis processes are well correlated.

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**ДОСЛІДЖЕННЯ КІНЕТИКИ ТЕРМІЧНОГО РОЗКЛАДАННЯ ДЕРЕВИНИ ЛИПИ З
ВИКОРИСТАННЯМ ТЕРМОГРАВІМЕТРИЧНОГО АНАЛІЗУ
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Анотація

У статті досліджено кінетику термічного розкладання деревини липи методом ТГА у повітрі при швидкості нагрівання 10–30 °С/хв. Визначено температурні діапазони та втрати маси для стадій дегідратації, піролізу і допалення вуглецевого залишку. Показано, що розкладання біомаси відбувається через розпад полімерних ланцюгів геміцелюлози, целюлози, лігніну, що входять до її складу. Вивчено вплив швидкості нагрівання на температуру максимуму втрати маси, яка становить 350–370 °С і відповідає розкладанню целюлози. Розраховано кінетичні параметри піролізу та для окремих стадій розкладання геміцелюлози й целюлози, використовуючи метод Коутса-Редферна для оцінки енергії активації та передеконенційного множника.

Ключові слова: біомаса, деревина липи, піроліз, леткі, коксовий залишок, TGA, хімічна кінетика, константа реакції, енергія активації.